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REFRACTORY-METAL-FIBER — NICKEL-BASE-ALLOY COMPOSITES FOR USE AT HIGH TEMPERATURES

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ABSTRACT

The stress-rupture properties of several refractory-metal-fiber - nickel-base-alloy composites were determined at 2000° and 2200° F (1093° and 1204° C). The effect of wire diameter, fiber content, fabrication history and compositions of the matrix and fiber on the stress-rupture properties of the composite was determined. The 100-hour stress-rupture strength obtained for the strongest composites tested at 2000° and 2200° F (1093° and 1204° C) was 35 000 and 14 000 psi (241.32 and 96.53 MN/m^2), respectively. Composite strength was related to the compatibility of the fibers with the matrix materials. Composite rupture strength can be optimized by proper fiber diameter selection.

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SUMMARY

An investigation was conducted to evaluate the stress-rupture properties of composites containing various combinations of refractory metal fibers and nickel-base alloys at 2000° and 2200° F (1093° and 1204° C). The effects of wire size, volume-fraction fiber content, and composition of the matrix and the fiber on the stress-rupture properties of the composite were determined.

Composites of refractory-metal fiber reinforced nickel-base alloys were produced having stress-rupture properties superior to conventional superalloys at use temperatures of 2000° and 2200° F (1093° and 1204° C). The 100-hour stress-rupture strength obtained for the best composites tested at 2000° and 2200° F (1093° and 1204° C) was 35 000 and 14 000 psi (241.32 and 96.53 MN/m^2), respectively.

Composite strength was related to the compatibility of the fibers with the matrix materials. Stronger composites were produced with matrix materials which reacted less with the fibers than those which were less compatible with the fibers. Nickel alloys containing titanium and aluminum additions appeared to be more compatible with the fibers investigated than nickel alloys which did not contain these additives. The refractory fiber composition also influenced the compatibility between the fiber and matrix. Commercially pure tungsten and tungsten - 3-percent-rhenium fibers were more compatible with the nickel alloys studied than were tungsten - 1 percent-thoria or TZM (a molybdenum alloy) fibers.

Fiber diameter was important to the design of composites in which reaction between the fiber and matrix material occurs. The stress-rupture strength of composites can be optimized by the proper fiber diameter selection. Generally, small-diameter fibers are more advantageous than large-diameter fibers for short-time stress-rupture applications. For long-time stress-rupture applications, however, large-diameter fibers are superior to small-diameter fibers. A graphical technique was used to illustrate schematically the variation of composite strength as a function of fiber diameter and depth of reaction of the fiber with the matrix.

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INTRODUCTION

Superalloys have recently been developed that have high strength at temperatures up to 1900° F (1038° C). There is, however, a need for high-strength materials at temperatures above 2000° F (1093° C) for applications such as advanced air-breathing engines. Such applications also require that the material have good oxidation resistance. Refractory-metal alloys with sufficient strength at these temperatures lack oxidation resistance.

Previous work at the Lewis Research Center has shown that refractory metal fibers have excellant high-temperature strength (ref. 1). Combinations of refractory-metal fibers and superalloys in the form of composites may be promising if advantage can be taken of the high strength of the refractory fiber and the good oxidation resistance of the superalloy. In model system studies conducted at Lewis, it was shown that composites had unusually high strength at elevated temperature as well as at room temperature. These composites obeyed a law of mixture relation for both tensile and stress-rupture conditions, (refs. 2 to 5). It was also shown that, when alloying reactions occurred between the fiber and the matrix, the composite properties were lowered. Preliminary compatibility studies have indicated that alloying reactions occurred between the fiber and the matrix in a refractory fiber reinforced superalloy composite (ref. 5).

Previous work has shown that the tensile and stress-rupture properties of conventional superalloys can be improved by reinforcement with refractory metal wires. Dean (ref. 6) recently reported on the reinforcement of cast conventional nickel-base superalloys with tungsten wires. Specimens containing up to 50-volume-percent fibers were tested in tension and stress-rupture at temperatures up to 2000° F (1093° C). A substantial improvement in the tensile strength and 100-hour stress-rupture strength of the nickel-base alloy was noted by the reinforcement of refractory-metal wires. A nickel-base alloy reinforced with 50-volume-percent tungsten wire had a 100-hour stress-rupture strength at 2000° F (1093° C) of 19 000 psi (131.00 MN/m^2). Baskey (ref. 7) has reported on the reinforcement of a nickel alloy with tungsten wires. The ultimate tensile strength of the nickel alloy reinforced with 33-volume-percent tungsten wires at 2000° F (1093° C) was four times that of the unreinforced material. However, both of these investigations have shown that the use of conventional nickel-base alloy material as a matrix material and current methods of making composites results in reactions with the fibers which are detrimental to the properties of the composite. Based on strength to density considerations at 2000° F (1093° C), these composite materials were little or no better than the best available superalloys. Nonetheless, refractory-metal-fiber reinforced superalloy matrix composites with superior strength to density properties would be possible if the loss of the fiber properties within the matrix could be limited.

The present investigation was conducted to produce fiber reinforced superalloy com-

posites having stress-rupture properties superior to conventional superalloys at use temperatures of 2000° and 2200° F (1093° and 1204° C). In addition, observations were made to relate variations in fiber-matrix compatibility to differences in matrix and fiber composition, fabrication procedure, wire diameter, test-time, and temperature. Four matrix compositions were chosen to be more compatible with the fibers than commercial superalloys. Nickel-base alloys containing varying percentages of refractory-element additions were vacuum cast and subsequently atomized into fine powders. The metal powder was slip cast around wire bundles of TZM (0.5-percent Ti - 0.08-percent Zr - 0.015-percent C - bal. Mo), 3D (tungsten - 3-percent rhenium), NF (tungsten - 1-percent thoria) and 218CS (commercial tungsten) wire bundles. The composites were sintered and isostatically hot pressed to produce fully dense composite specimens containing up to 70-volume-percent fibers. Compatibility among the various combinations of matrix materials and wire was studied at 2000° and 2200° F (1093° and 1204° C) for 100 hours. Stress-rupture data at 2000° and 2200° F (1093° and 1204° C) were obtained for the refractory-metal fiber composites, unreinforced nickel-base alloys, and refractory-alloy wires.

MATERIALS, APPARATUS, AND PROCEDURE

Wire Material

The wire materials selected for use in this investigation were TZM, NF, 3D, and 218CS. Most of the data were obtained from specimens containing 0.008-inch (0.020-cm) diameter wires in the various matrices. As will be described subsequently, the stress-rupture results with this wire suggested that work be done with larger diameter wires. For these experiments, 0.015- and 0.020-inch (0.038- and 0.051-cm) diameter wires of 218CS and NF wires were used. The wire was received in the as-drawn, cleaned, and straightened condition. A chemical analysis made on the NF and 3D wire revealed that the NF wire contained 0.8 to 1.1 percent thoria and that the 3D wire contained 2.79 percent rhenium.

Nickel Alloys

The compositions of the nickel alloys were formulated based on forgeability, compatibility with the reinforcing fibers, and oxidation resistance at 2000° F (1093° C). A high percentage of refractory-metal was added to nickel to lower the reactivity with the reinforcing fibers by reducing the chemical potential differential for diffusion, and a high per-

centage of chromium was added to enhance oxidation resistance. Workability and oxidation resistance have been demonstrated for additions of up to 50-percent chromium and tungsten to nickel (ref. 8). A nickel alloy containing 20-percent chromium and 25-percent tungsten was one of the alloys investigated, as shown in table I. This basic composition was modified to improve strength and compatibility. Aluminum additions were made to form a gamma prime phase (Ni_3Al), and titanium additions to form an eta phase (Ni_3Ti) both of which precipitation harden the alloy. The additions to the initial alloy were substituted for corresponding amounts of chromium. These additions tie up three atoms of nickel for each atom added and further lower the reactivity of the matrix alloy with the

TABLE I. - SELECTED NICKEL ALLOY

MATRIX MATERIALS

Alloy	Nominal composition of alloy, wt %							
	Al	Cb	Cr	Mo	Ni	Ti	W	Ta
1	---	----	20	--	55	---	25	----
3	2	----	15	--	56	2	25	----
5	---	1.25	19	4	70.5	---	4	1.25
7	4.2	1.25	15	4	66.8	3.5	4	1.25

TABLE II. - CHEMICAL ANALYSIS OF NICKEL-ALLOY

METAL POWDERS

Alloy	Composition, wt %						
	Al	C	Cr	Cb	Mo	P	S
1	----	0.0041	19.50	----	----	0.0012	<0.001
3	1.96	.0032	15.19	----	----	.0006	<.001
5	----	.0037	18.59	1.24	3.92	.0015	<.001
7	4.15	.0029	14.86	1.18	3.95	.0010	<.001

Alloy	Composition, wt %						
	Ti	Ta	W	N_2	O_2	H_2	Ni
1	----	----	24.75	0.0051	0.1400	0.0026	bal.
3	1.84	----	24.61	.0100	.0063	.0020	bal.
5	----	1.25	4.30	.0072	.1100	.0022	bal.
7	3.41	1.30	4.33	.0039	.5300	.0027	bal.

TABLE III. - MEASURED DENSITIES OF VACUUM-CAST NICKEL-BASE ALLOYS

Alloy	Density	
	g/cc	lb/in.^{3}
1	9.67	0.349
3	9.15	.330
5	8.72	.315
7	8.09	.292

fiber by lowering the nickel potential for diffusion. The composition of this alloy is also shown in table I. The two other alloys selected are shown in table I and had compositions similar to some conventional nickel-base alloys with good workability and oxidation resistance. They also have high-refractory-metal contents to promote compatibility with the fibers. The second of these alloys differs from the first in that it contains aluminum and titanium additions, again substituted for chromium.

The nickel alloys were vacuum cast and atomized into fine powders. A chemical analysis of the powder is shown in table II. Vacuum cast stress-rupture specimens for each alloy composition were obtained from the master melt used for making the powder. The densities of the alloys are tabulated in table III and are based on the measurements of 12 specimens of each alloy composition.

Composite Specimen Fabrication

Slip composition. - An ammonium salt of alginic acid was selected as the binder material for the slips studied in this investigation. In previous work on the slip casting of several nickel-base alloys using this material as a binder (ref. 9) densities on the order of 95 percent of theoretical were obtained for sintering temperatures of 2300° F (1260° C). The binder material is water soluble and has a low viscosity: a 1-percent solution has a viscosity of 85 centipoises (0.085 (N)(sec)/m^2) at 76° F (25° C). It decomposes at 410° to 430° F (210° to 225° C) by carbonizing. The residual carbon can be burned off completely at 1250° F (677° C). The ash content is 4 percent maximum. In previous work with this binder material (ref. 9), no noticeable contaminants could be detected after sintering.

In the present work, a 2.5-percent solution of the binder material in water was used. This solution was then added to the metal powder, having a particle size of -325 to +500 mesh, and then diluted with water so that the solid to liquid ratio and viscosity of the

TABLE IV. - METAL POWDER SLIP COMPOSITIONS AND PROPERTIES

Alloy	Composition, wt %			Viscosity at infinite shear		Slip density, g/cc	Percent theoretical density of slip casting	pH
	Metal powder	Water	Binder material	cP	$(N)(sec)/m^2$			
1	90.90	8.98	0.12	1500	1.5	5.8	60	7.4
3	89.90	10.00	.10	3000	3.0	5.2	57	7.4
5	90.90	8.98	.12	5500	5.5	5.3	61	7.3
7	80.80	18.97	.23	3000	3.0	4.6	57	7.6

slip was lowered to the point where the slip was pourable. The viscosity of the slip was less than 5000 centipoises (5.0 $(N)(sec)/m^2$). Viscosity of the slips was measured with a viscometer at spindle speeds of 6, 12, and 30 rpm. Measurements of the pH of the slips were made using a pH meter.

The metal powder content, water content, and binder content used for each alloy system are listed in table IV. Between 80 and 90 percent by weight of metal powder is contained in the slip.

The fluidity of metal slips can often be greatly increased by adjusting the pH of the slip. The most dense slips for a predetermined viscosity are obtained at a specific pH value. Studies were conducted to determine whether the viscosity of the slip used could be reduced by changing the pH of the slip. Changes in the pH of the slip were made by adding either sodium hydroxide or nitric acid to the slip. The viscosity of the slip was then measured. The viscosity of the slip without the acidic or basic additions was about the minimum that could be obtained.

Composite fabrication procedure. - Composites were made using -325 to +500 mesh powder and having the slip compositions shown in table IV. Continuous-length refractory-wire bundles were inserted into a nickel tube containing a wire screen at the bottom and several layers of filter paper as shown in figure 1. The nickel tube was connected to a rubber hose that was attached to a mechanical pump. The nickel tube was then placed on a vibrating table and slip was poured into the wire bundle while vibrating the tube. As the nickel-alloy powder settled to the bottom of the bundle excess liquid media was siphoned off the top and more slip was added. This process was continued until the nickel-alloy powder completely infiltrated to the top of the wire bundle. The vibrator was then turned off and a vacuum was applied to the tube to drive off any additional liquid media left in the casting. The specimen was removed from the tube and dried in air for approximately 24 hours at 140° F (60° C). The specimens were then processed by either a high-temperature-densification technique or a low-temperature-densification

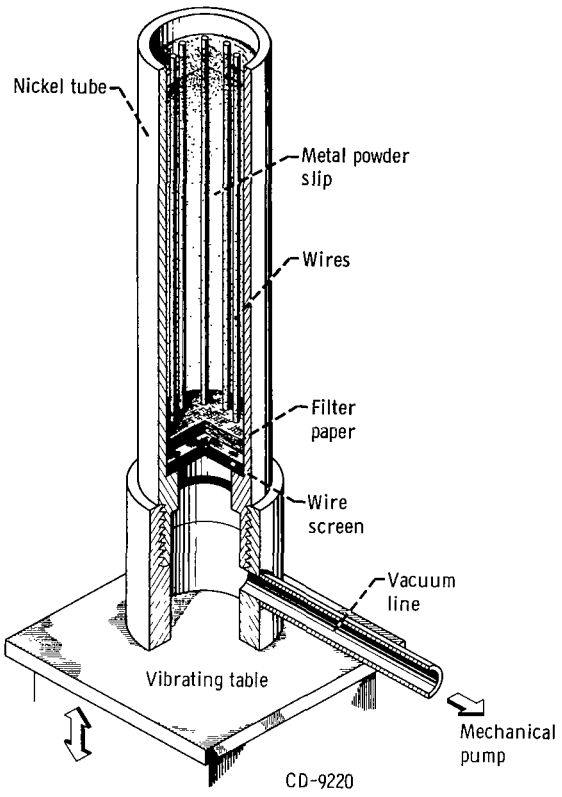


Figure 1. - Slip casting apparatus.

technique. Initially, the high-temperature-densification technique was used. The high-temperature-densification technique consisted of sintering the slip cast specimen at 2000° F (1093° C) for 1 hour in dry hydrogen to drive off the binder and to reduce any nickel or chromium oxide film that might be present on the surface of the powders. Nickel oxide is readily reduced at this temperature using hydrogen. The reduction of chromium oxide, however, requires very dry hydrogen. The dew point of the hydrogen used in this investigation was -88° F (-67° C), which should be sufficient to reduce any chromium oxide film contained on the powders (ref. 10). Any titanium or aluminum oxide film present, however, would not be reduced (ref. 10). After sintering, the specimens were inserted into closely fitting nickel cans having a wall thickness of 0.030 inch (0.076 cm). Nickel plugs were inserted into the top and bottom of the can, and the can was electron beam welded in a vacuum. The cans were then leak tested in helium. Final densification was accomplished by isostatically hot pressing the canned billet at 2000° F (1093° C) for 2 hours under helium pressurized to 20 000 psi (137.89 MN/m^2). Later, in the program, the densification technique was modified to a low-temperature-densification technique. The low-temperature-densification technique followed the same

procedure as that used in the high-temperature-densification technique. The slip cast specimens, however, were sintered at 1500° F (816° C) for 1 hour in dry hydrogen rather than at 2000° F (1093° C). Final densification was accomplished by isostatically hot pressing the billets first at 1500° F (816° C) for 1 hour, and then 2000° F (1093° C) for 1 hour under helium pressurized to 20 000 psi (137.89 MN/m^2).

Stress-Rupture Tests

Stress-rupture tests on single fibers were conducted in a stress-rupture apparatus specifically designed for the testing of up to 4 filaments simultaneously. A detailed description of this apparatus may be found in reference 1. A photograph of the inside of the chamber is shown in figure 2. In this testing unit, the wire was strung through a tantalum-wound resistance furnace and around a pulley and attached to a weight pan. The chamber was closed, and the system was evacuated to a measured vacuum of approximately 5×10^{-5} to 1×10^{-6} torr (6.65×10^{-3} to 1.33×10^{-4} N/m^2). The furnaces were then turned on and allowed to stabilize at the desired test temperature. After stabilization the weights were applied to the specimens by lowering the retractable support. Tests were conducted at 2000° and 2200° F (1093° and 1204° C) for periods up to 200 hours.

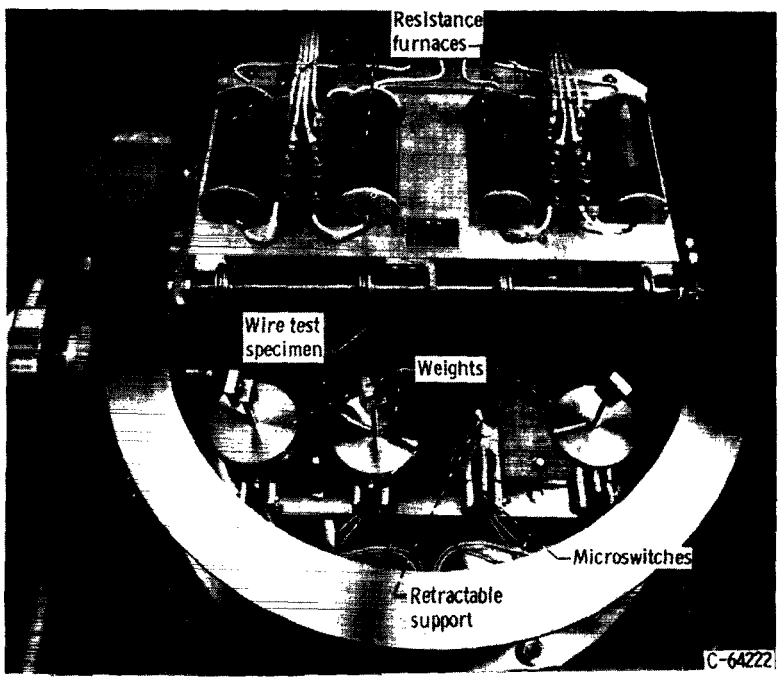


Figure 2. - Fiber stress-rupture testing apparatus.

Stress-rupture tests on vacuum-cast nickel-alloy specimens and on composite test specimens were conducted in conventional creep machines, using a helium atmosphere to limit oxidation. Tests were conducted at 2000° F (1093° C) and, in some cases, 2200° F (1204° C).

Compatibility Studies

As-drawn, cleaned, and straightened wires of NF, 218CS, 3D, and TZM were annealed at 2000° F (1093° C) in helium for 100 hours. The transverse and longitudinal sections were examined metallographically to determine the effect of the annealing treatment on the microstructure of the annealed wire without the influence of a matrix.

Compatibility studies were conducted on all combinations of matrix and fiber materials. Compatibility specimens were slip cast and then fabricated by the high-temperature-densification technique. Specimens having dimensions of 0.75-inch (1.91-cm) diameter by 0.50-inch (1.47-cm) length were then cut from the pressed billets. The microstructure of the fiber-matrix interface was examined metallographically, and the depth of reaction was measured optically on transverse sections of composite specimens using a magnification of 500. The depth of the reaction zone was defined as the distance from the fiber-matrix interface to the interface in the fiber where a microstructural change was observed. The effect of time and temperature on fiber matrix compatibility was studied using failed stress-rupture specimens fabricated by either the high- or low-temperature-densification technique.

Electron Microprobe Studies

Electron microprobe studies were conducted on transverse sections of composite specimens fabricated by the high-temperature-densification technique. These studies were made to determine whether there was elemental diffusion between the refractory wires and the matrix and to try to identify these elements and the extent to which they diffused. A step scan mode (1-μm per step), was used in the analysis.

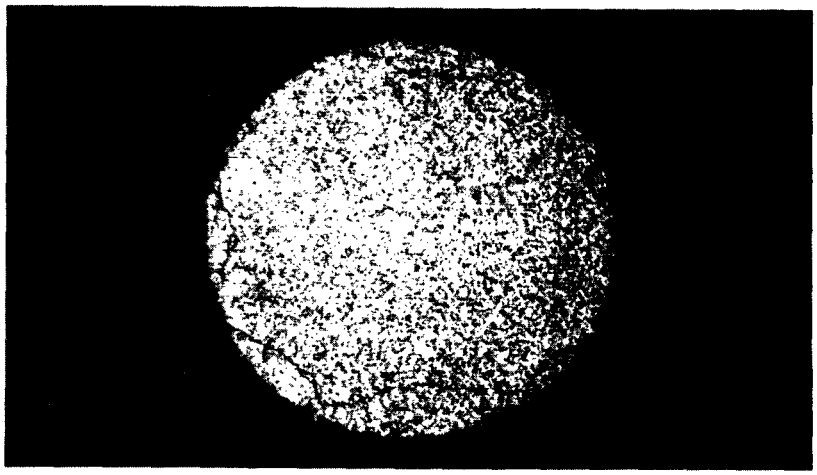
RESULTS

The results of this investigation showed that the high-refractory-metal content matrix materials selected were sufficiently compatible with the wire reinforcement to limit fiber property loss. A fabrication procedure was evolved which achieved greater

than 99-percent densification of composites at a low sintering temperature. Composites with excellent properties were obtained by limiting the reaction between fiber and matrix to a depth of 1.5 mils (0.0038 cm) or less for exposure times of 100 hours at 2000° F (1093° C). The data collected to obtain these composite properties will now be described.

Wire Anneal Studies

Photomicrographs showing the microstructure of the annealed wires are shown in figure 3. The wires still maintained a fibrous structure. The thermal treatment for

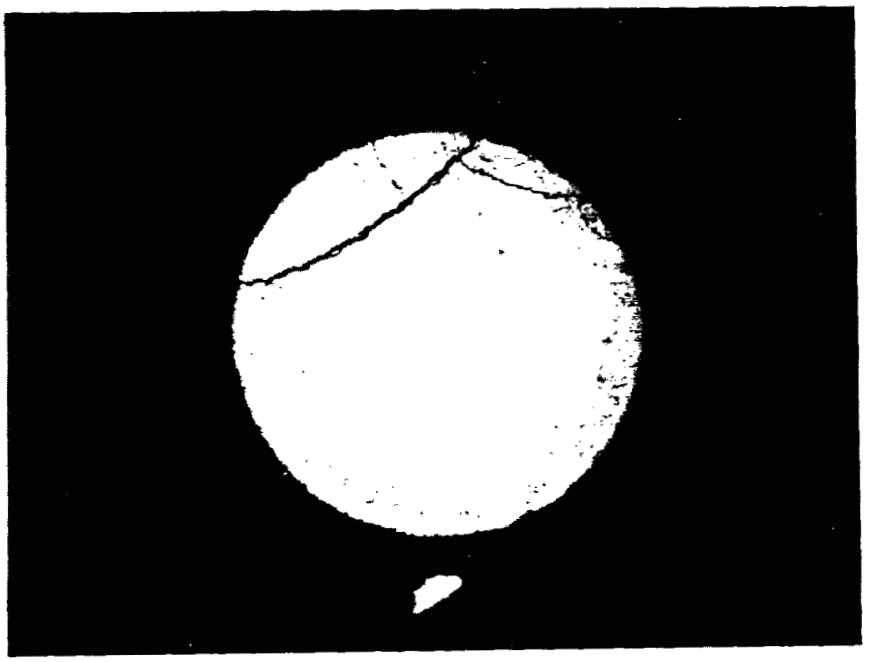


Transverse section

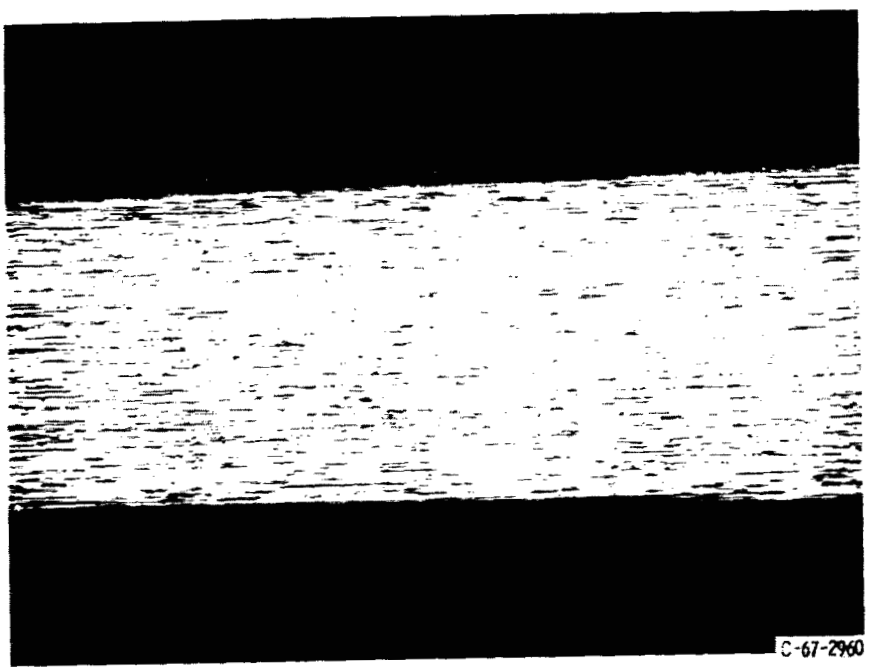
Longitudinal section

(a) 218CS alloy wire.

Figure 3. - Microstructure of wire-annealed 100 hours at 2000° F (1093° C) in helium. X250.



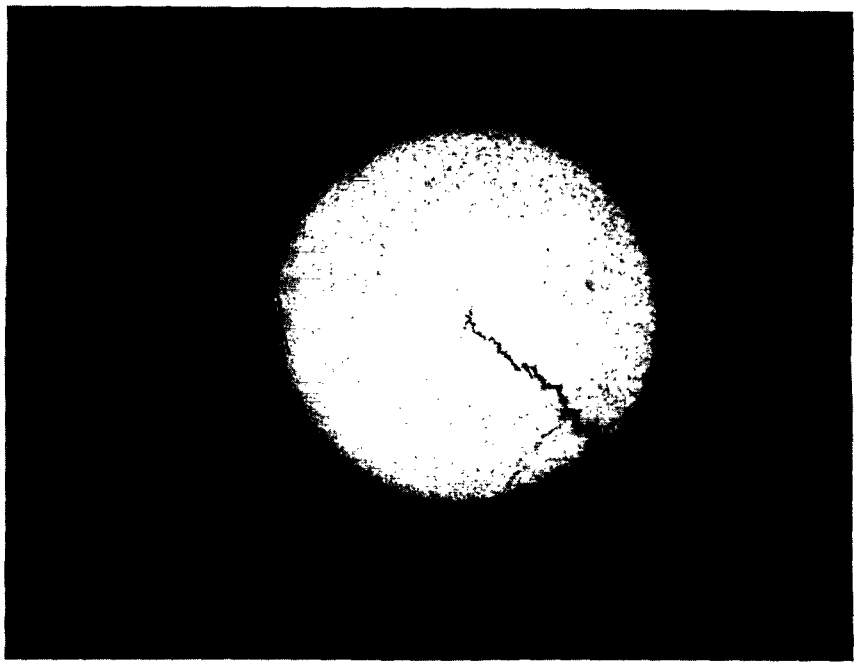
Transverse section



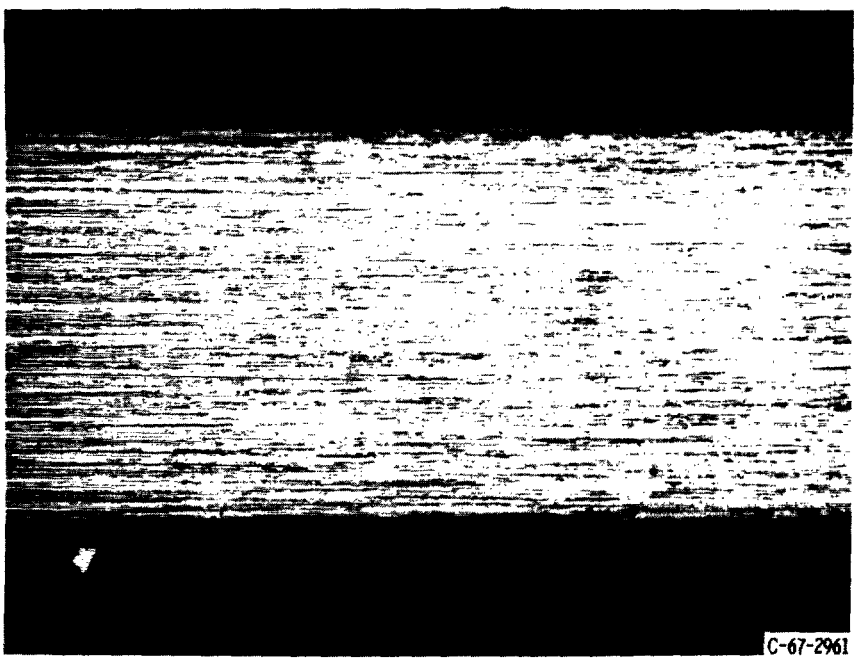
Longitudinal section

(b) TZM alloy wire.

Figure 3. - Continued.



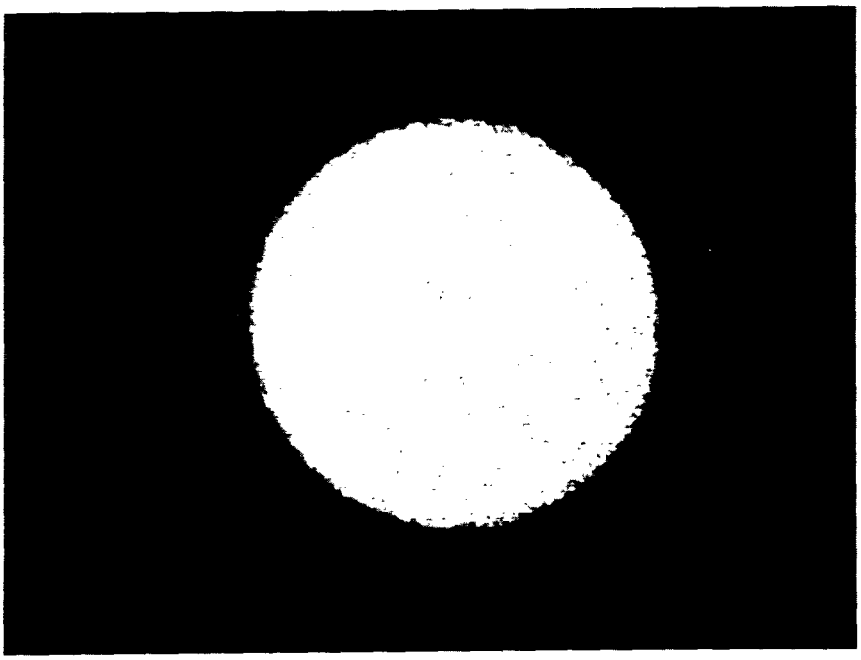
Transverse section



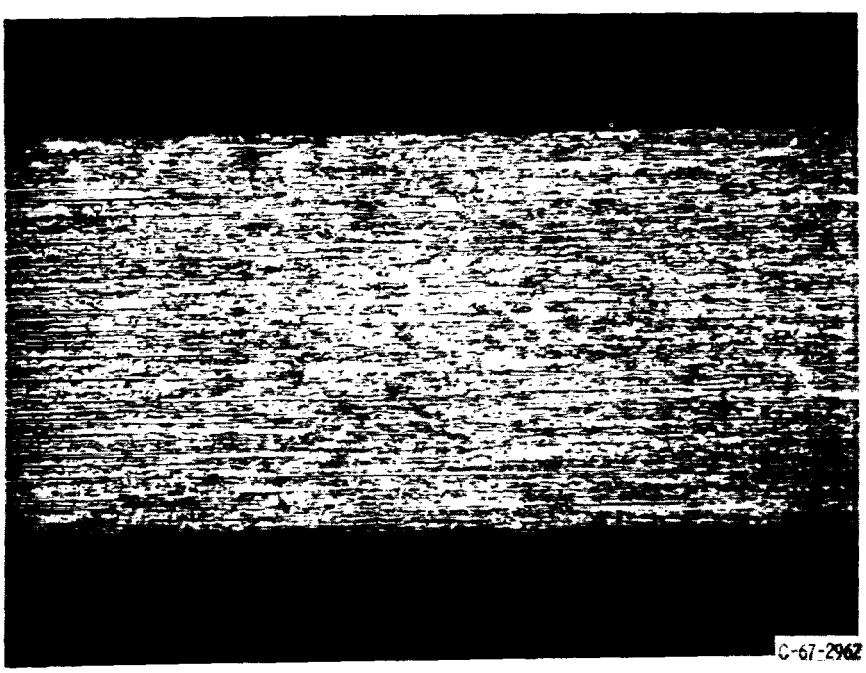
Longitudinal section

(c) 3D alloy wire.

Figure 3. - Continued.



Transverse section



Longitudinal section

(d) NF alloy wire.

Figure 3. - Concluded.

100 hours at 2000° F (1093° C) did not cause a severe change in microstructure. Cracks appearing in the wires are a result of specimen preparation.

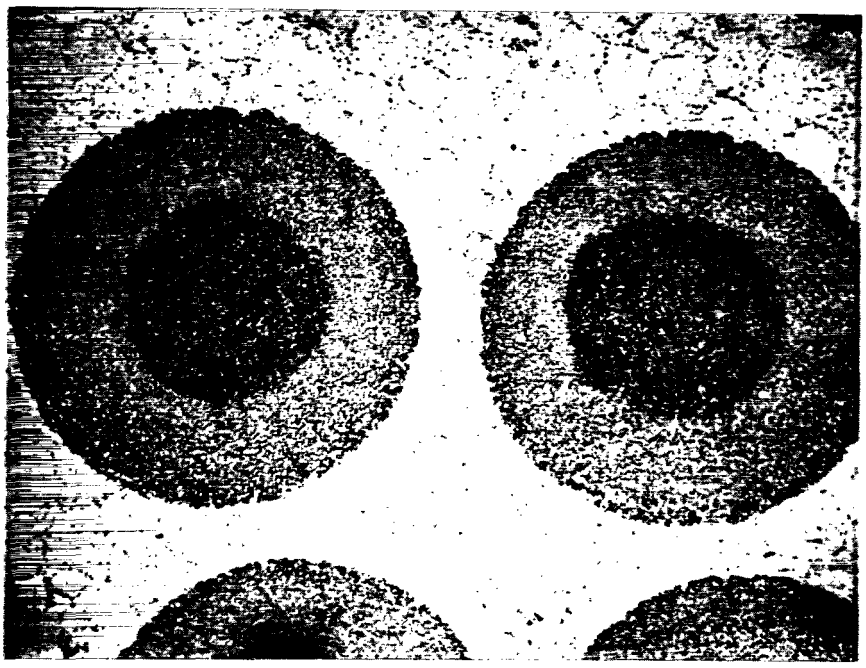
Composite Compatibility Studies

Several microstructures typical of as-sintered and pressed composite specimens containing 218CS or TZM wire are shown in figure 4. The figure shows the relative effects of the reaction of the matrix with the fiber. Alloys 3 and 7, which contain aluminum and titanium, are more compatible with the fibers than alloys 1 and 5. Alloys 1 and 5 do not contain aluminum or titanium. It can also be seen that the TZM wires show severe reaction with the matrix materials and are completely recrystallized. The visible depth of penetration of the nickel alloy into the refractory wires was measured and is shown in table V. The table shows that the 218CS and 3D wire are more compatible with the alloys than the other two wire materials investigated.

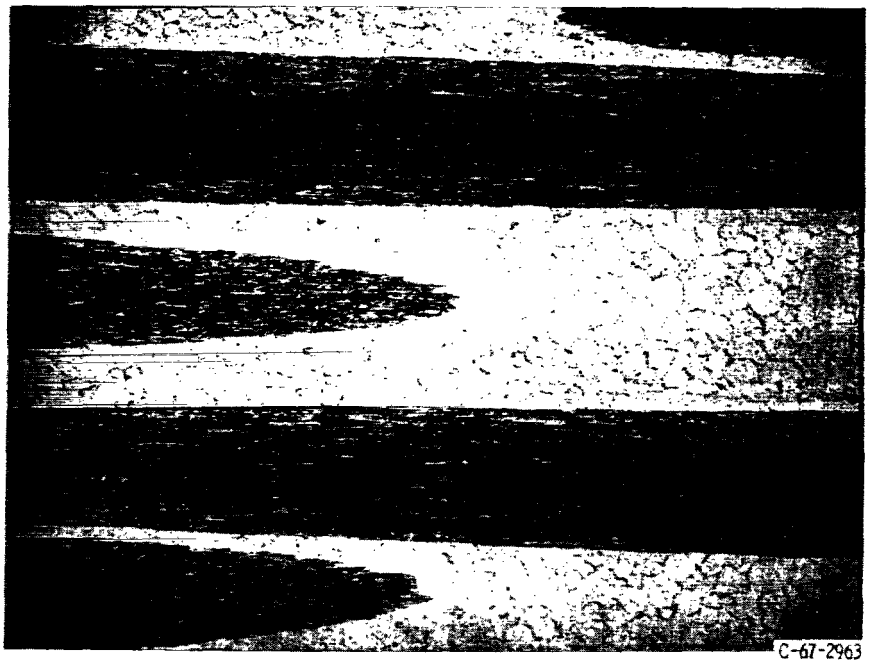
TABLE V. - COMPATIBILITY STUDIES OF AS-PRESSED SPECIMENS

[Fabricated by high-temperature-densification technique.]

Alloy	Wire material	Depth of penetration	
		in.	cm
1	218CS	0.00200	0.00508
	NF	.00250	.00635
	3D	.00150	.00381
	TZM	Complete	Complete
3	218CS	0.00100	0.00254
	NF	.00180	.00457
	3D	.00100	.00254
	TZM	Complete	Complete
5	218CS	0.00300	0.00762
	NF	.00325	.00825
	3D	.00320	.00812
	TZM	Complete	Complete
7	218CS	0.00075	0.00190
	NF	.00150	.00381
	3D	.00100	.00254
	TZM	Complete	Complete



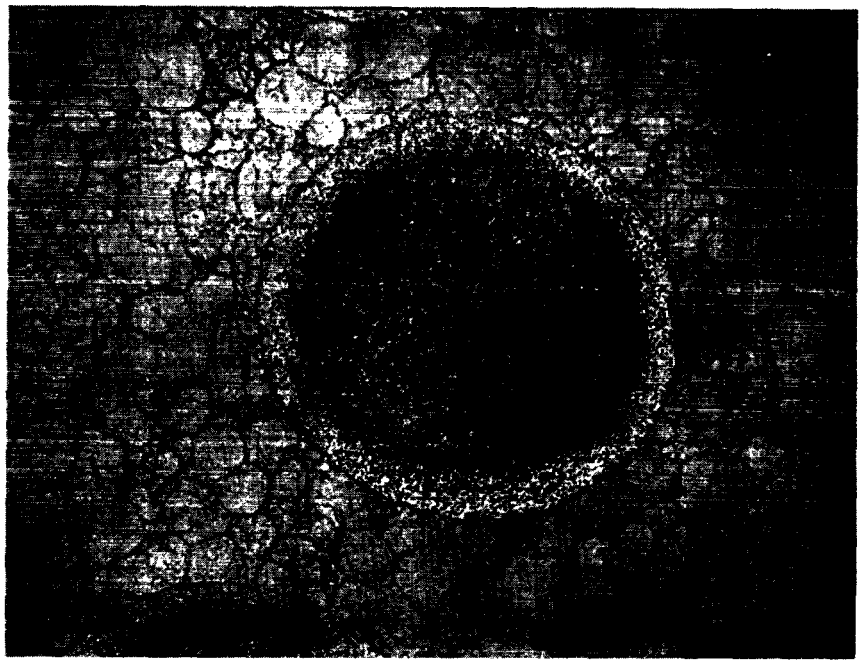
Transverse section. X250.



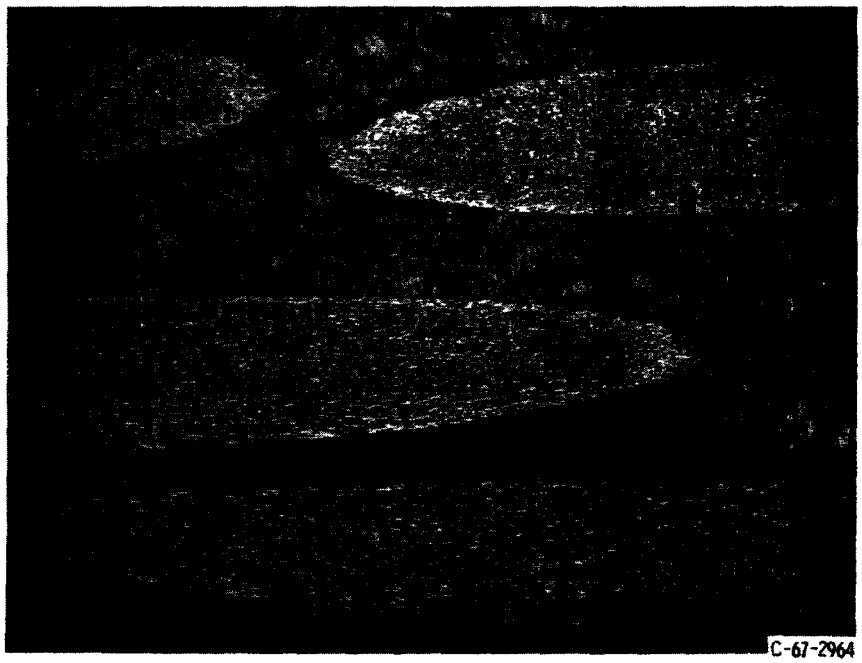
Longitudinal section. X100.

(a) Alloy 1 (Ni, W, Cr plus 218CS wire composite.

Figure 4. - Microstructure of high-temperature fabricated composites (sintered 1 hour at 2000° F, (1093° C); hot pressed 2 hours at 2000° F (1093° C); pressure, 20 000 psi (137.89 MN/ni^2).



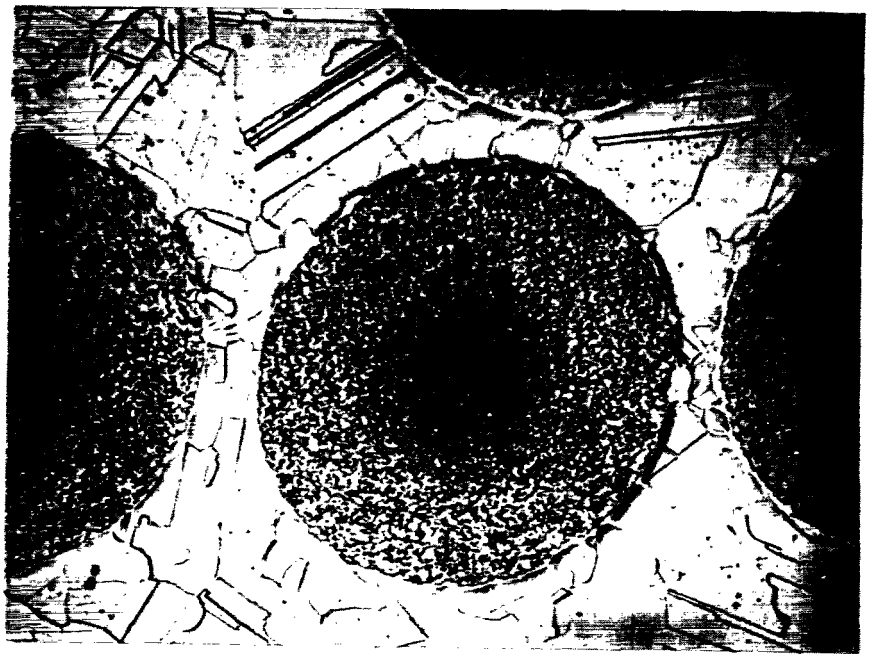
Transverse section. X250.



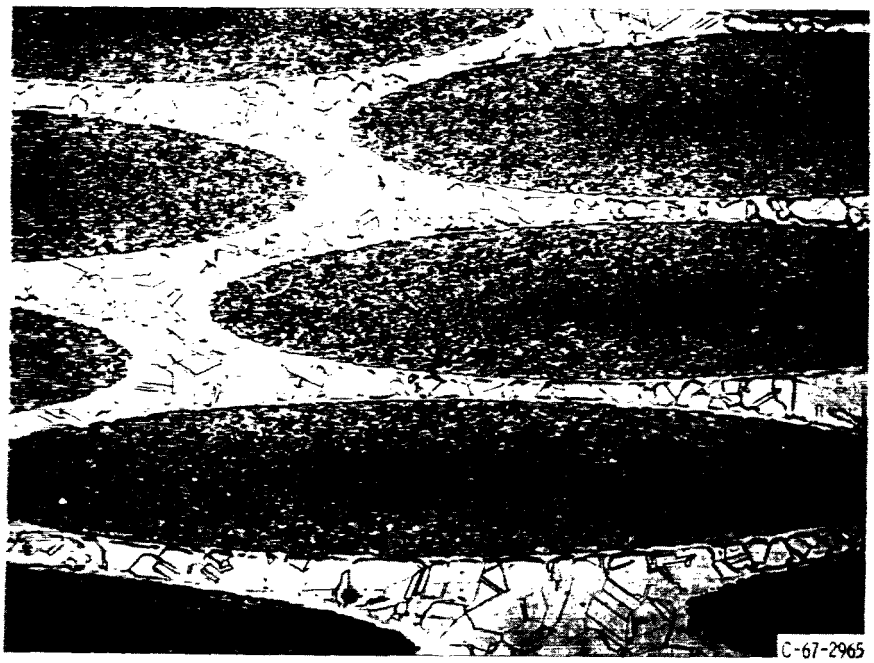
Longitudinal section. X100.

(b) Alloy 3 (Ni, W, Cr, Ti, Al) plus 218CS wire composite.

Figure 4. - Continued.



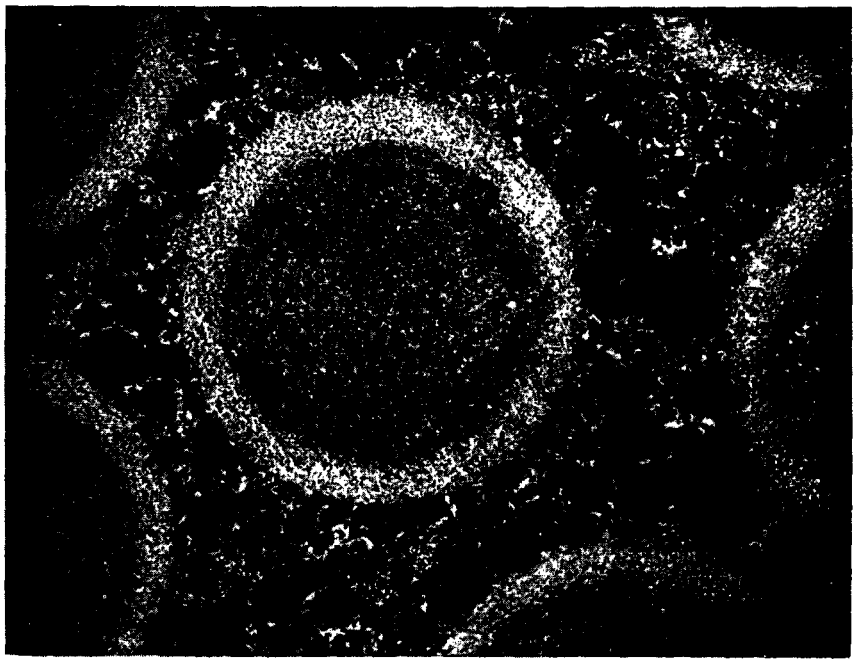
Transverse section. X250.



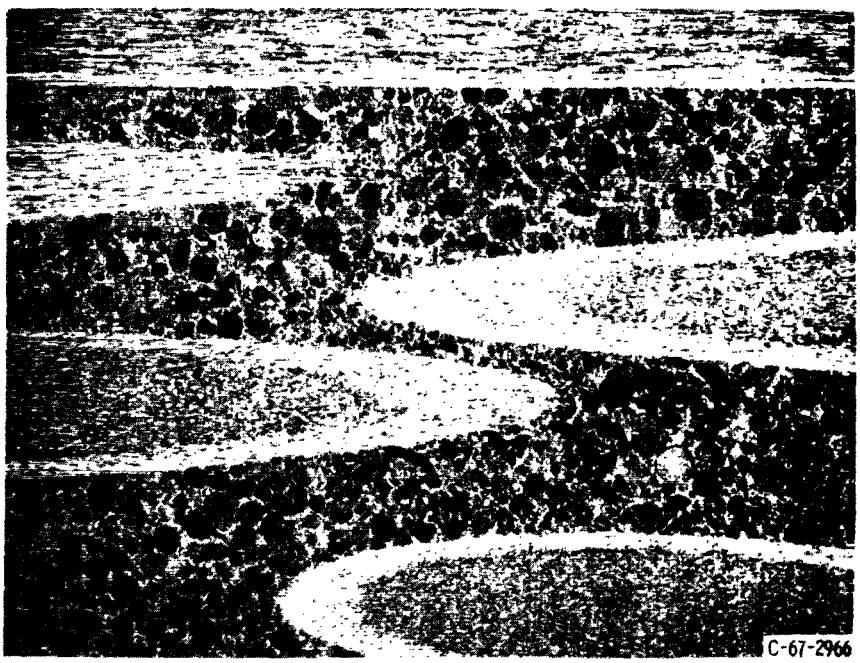
Longitudinal section. X100.

(c) Alloy 5 (Ni, W, Cr, Mo, Cb, Ta) plus 218CS wire composite.

Figure 4. - Continued.



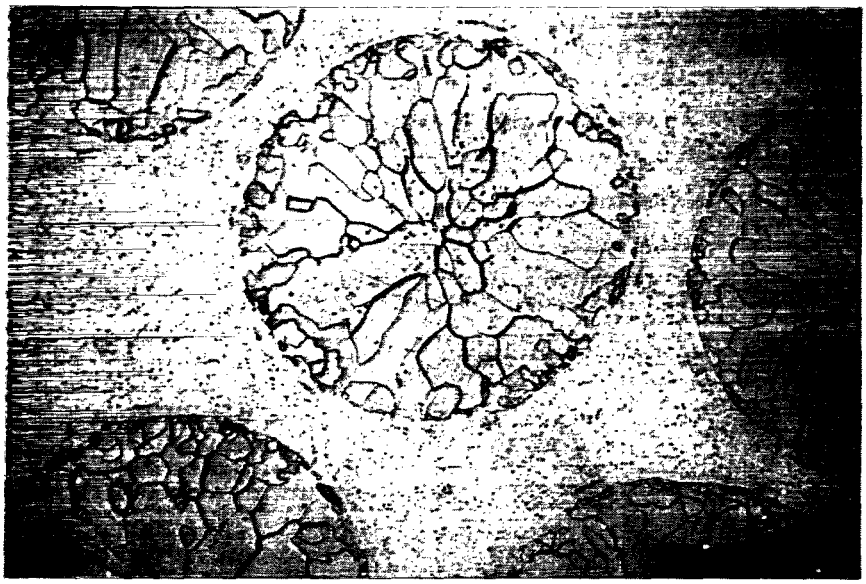
Transverse section. X250.



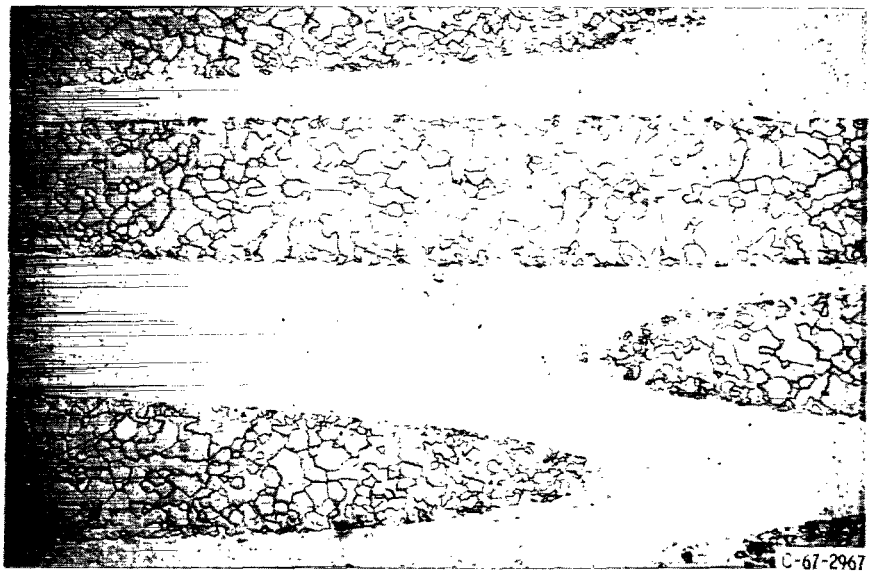
Longitudinal section. X100.

(d) Alloy 7 (Ni, W, Cr, Mo, Cb, Ta, Ti, Al) plus 218CS wire composite.

Figure 4. - Continued.



Transverse section. X250.



Longitudinal section. X100.

...er Alloy I (Ni, W, Cr plus TZM wire composite.

Figure 4. - Concluded.

Electron Microprobe Study

The results of the electron microprobe scans showed detectible diffusion zones that were of the order of 0.25 mil (0.0006 cm) to a maximum of 0.50 mil (0.00128 cm) in depth. Optical measurements, however, indicated that the depth of the zones were on the order of 2 mils (0.0051 cm). It is difficult for the probe to detect element contents below 1 percent. The greater portion of the diffusion zone must, therefore, contain less than 1 percent of the duffusing elements. All the specimens indicated some diffusion of tungsten into the matrix although the greater depth of the diffusion was into the tungsten wire. There was subtle evidence from the microprobe study that grain boundary diffusion occurred, which may explain the difference in depth of the zone determined optically as compared with the depth determined by the probe. A typical concentration against distance plot for 218CS wire in alloy 3 is shown in figure 5.

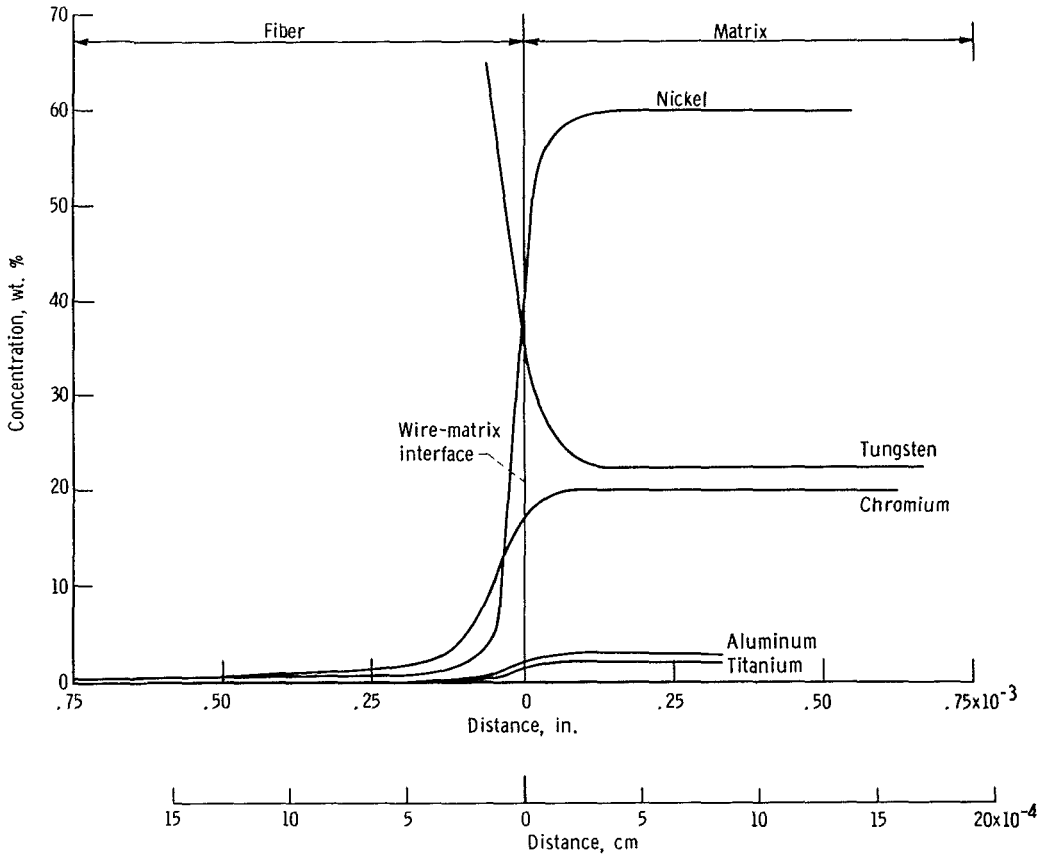
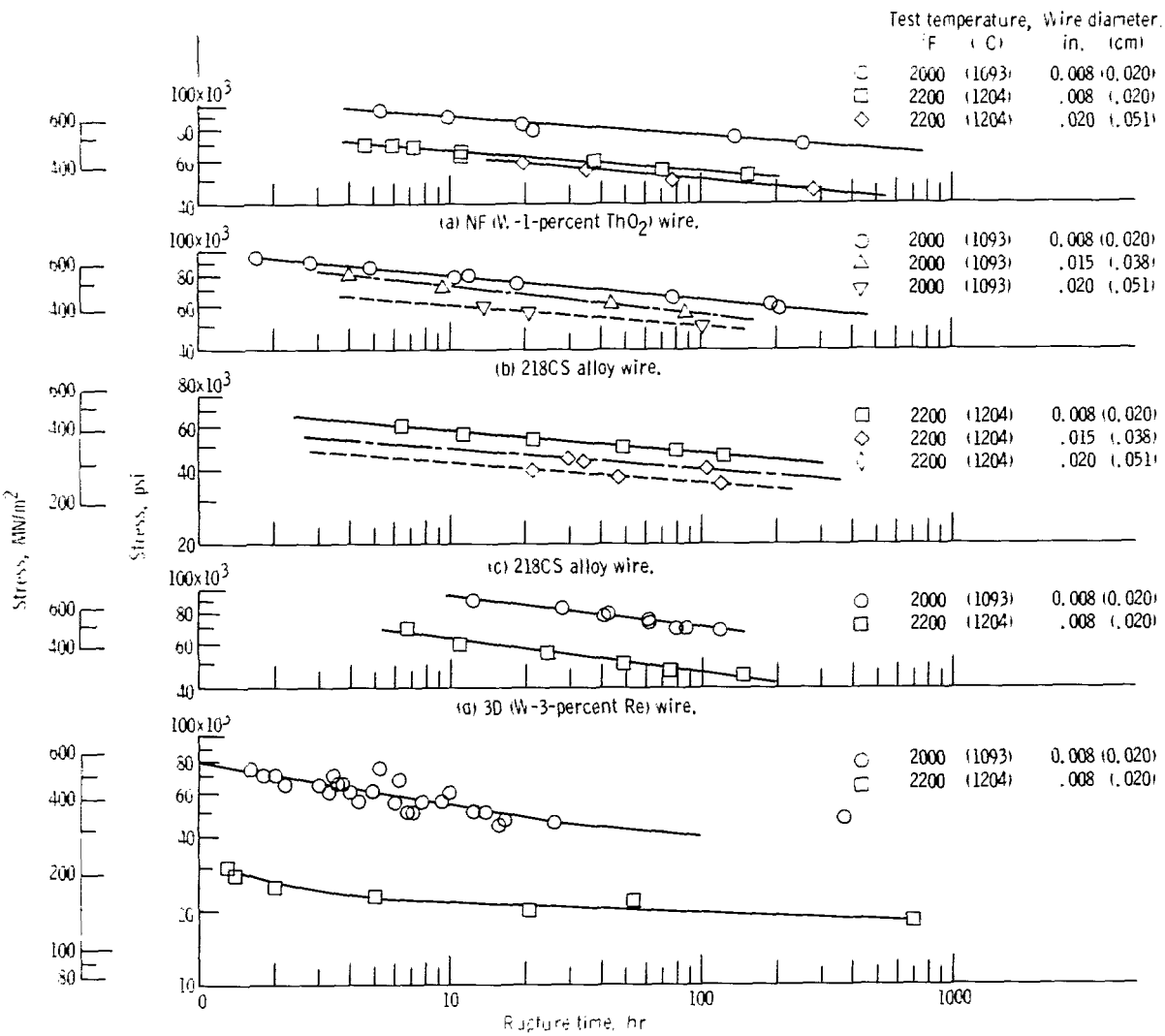


Figure 5. - Typical concentration versus distance plot determined by microprobe.

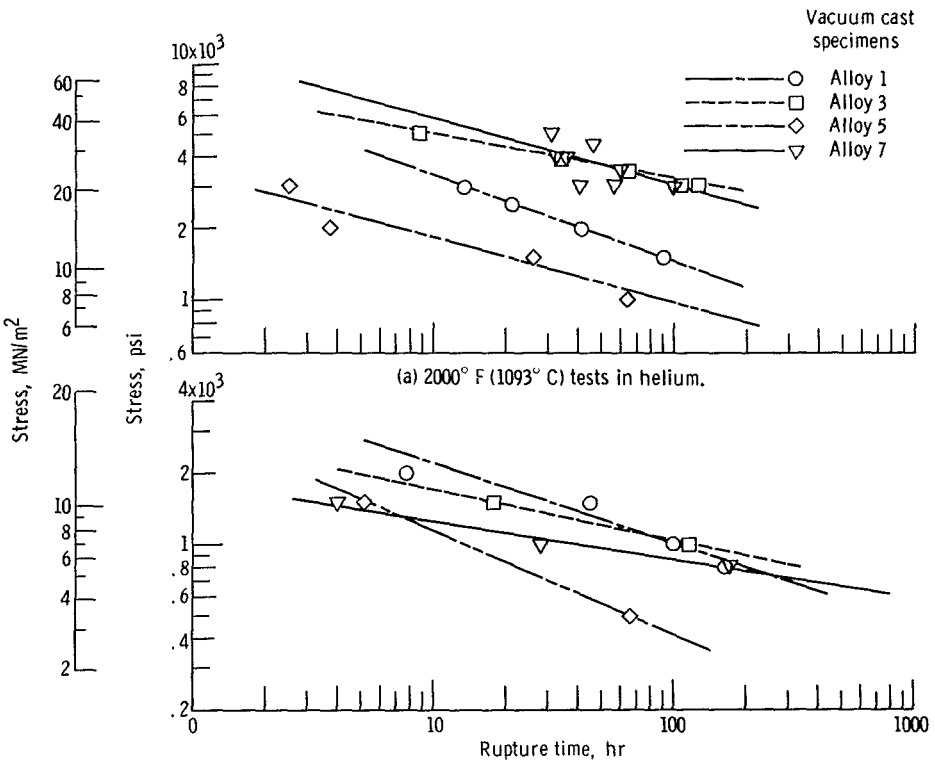
Stress-Rupture Results

The stress to cause rupture against rupture life at 2000° and 2200° F (1093° and 1204° C) for the wire materials studied is plotted in figure 6. The scatter in the stress-rupture data is small for the tungsten alloys but is quite large for the TZM wire at 2000° F (1093° C) (fig. 6(e)). The stress-rupture properties of the 218CS wire decreased with increasing wire diameter (figs. 6(b) and (c)). The same is true for the NF wire, however, the decrease in properties is much less (fig. 6(a)). The NF wire was the strongest wire material investigated. In fact, the 1000-hour stress-rupture strength of



(e) TZM (Mo-0.5-percent Ti - 0.08-percent Zr - 0.015-percent C) wire.

Figure 6. - Wire rupture properties. All tests in 1×10^{-5} torr (1.33×10^{-3} N/m^2) vacuum.



(a) 2000° F (1093° C) tests in helium.

(b) 2200° F (1204° C) tests in helium.

Figure 7. - Matrix alloy rupture properties.

the NF wire is equivalent to the 100-hour stress-rupture strength of the 218CS and 3D wire.

Similar plots for the unreinforced nickel alloys tested at the same temperatures are shown in figure 7. The nickel alloys containing the titanium and aluminum additions (alloys 3 and 7) are stronger at 2000° F (1093° C) than alloys 1 and 5, which do not contain these additions. At 2200° F (1204° C) alloys 1 and 3, which contain the higher percentages of refractory additions, are the stronger materials.

The stress to cause rupture in 100 hours at 2000° and 2200° F (1093° and 1204° C) for the wire material and the nickel alloys is given in table VI. Also shown in the table is the stress to cause rupture in 100 hours divided by the material density (specific strength). At 2000° F (1093° C) the NF wire is the strongest wire material in stress-rupture; the TZM wire is almost as strong. At 2200° F (1204° C), however, the TZM wire is much weaker than the other wire materials studied. The strongest nickel alloy in stress-rupture for 100 hours is alloy 3 at both temperatures.

The results of the compatibility studies indicated that the 218CS wire and 3D wire materials were more compatible with the nickel alloys than the other wire materials studied. The 218CS wire material was selected as the reinforcement material for

TABLE VI. - STRESS-RUPTURE IN 100 HOURS FOR WIRE AND NICKEL ALLOYS

[From figs. 6 and 7.]

(a) Wire

Test temperature		Wire material	Wire diameter		Stress		Stress to density ratio	
°F	°C		in.	cm	psi	MN/m^2	in.	m
2000	1093	NF	0.008	0.020	76 000	524.00	110 000	2794.0
		3D	.008	.020	70 000	482.63	101 000	2565.4
		218CS	.008	.020	64 000	441.26	92 000	2336.8
		218CS	.015	.038	55 000	379.21	79 000	2006.6
		218CS	.020	.051	50 000	344.74	72 000	1828.8
		TZM	.008	.020	40 000	275.79	109 000	2768.6
2200	1204	NF	0.008	0.020	54 000	372.32	77 000	1955.8
		NF	.020	.051	50 000	344.74	72 000	1828.8
		3D	.008	.020	46 000	317.16	66 000	1676.4
		218CS	.008	.020	46 000	317.16	66 000	1676.4
		218CS	.015	.038	40 000	275.79	58 000	1473.2
		218CS	.020	.051	35 000	241.32	51 000	1295.4
		TZM	.008	.020	20 000	137.89	55 000	1397.0

(b) Nickel alloys

Test temperature		Alloy	Stress		Stress to density ratio	
°F	°C		psi	MN/m^2	in.	m
2000	1093	1	1500	10.34	4 300	109.2
		3	3300	22.75	10 000	254.0
		5	1000	6.89	3 200	81.3
		7	3000	20.68	10 200	259.1
2200	1204	1	1000	6.89	2 900	73.7
		3	1000	6.89	3 000	76.2
		5	400	2.76	1 300	33.0
		7	870	6.00	2 980	75.7

TABLE VII. - STRESS-RUPTURE PROPERTIES OF COMPOSITES

TESTED AT 2000° F (1093° C) IN HELIUM

[Fabricated by high-temperature-densification technique.]

Alloy	218CS wire diameter		Stress		Type of fracture (a)	Life, hr	Fiber content, vol. %
	in.	cm	psi	MN/m^2			
1	0.008	0.020	15 000	103.42	T	39.1	35.1
			15 000	103.42		51.0	33.2
			15 000	103.42		142.7	44.6
			20 000	137.89		16.7	29.1
			↓	↓		30.9	41.0
						35.0	37.7
						43.4	42.9
			25 000	172.37	S	7.9	37.7
3	0.008	0.020	15 000	103.42	T	0.2	11.6
			15 000	103.42	T	9.4	26.1
			15 000	103.42	T	68.2	33.6
			18 000	124.11	S	25.2	41.6
5	0.008	0.020	15 000	103.42	T	4.5	26.5
			↓	↓		5.0	21.1
						7.4	21.3
						13.6	33.4
						72.6	43.0
						86.8	46.2
			20 000	137.89		17.3	44.7
			20 000	137.89		29.4	41.1
7	0.008	0.020	15 000	103.42	T	4.5	16.5
			↓	↓	R	23.0	24.0
					T	61.2	33.8
					R	63.4	31.4
			20 000	137.89	R	72.4	44.1
3	0.015	0.038	15 000	103.42	T	246.6	34.1
			15 000	103.42	T	319.9	44.3
			20 000	137.89	S	95.7	55.8
	0.020	0.051	15 000	103.42	T	285.3	41.6
			20 000	137.89	S	45.5	52.6

[a] T, tensile failure; S shear failure; and R, failure at the radius.

stress-rupture studies of composites. The composites were fabricated using the high-temperature-densification technique. The stress-rupture properties of all the nickel alloys reinforced with 218CS wire were determined at 2000° F (1093° C) and are given in table VII. A number of composites failed by a shear fracture which resulted from the matrix shearing because of misalinement of the fibers with the test axis. The type of fracture which occurred is also listed in the table. A plot of fiber content against rupture life for specimens tested at 2000° F (1093° C) and 15 000 psi (103.42 MN/m^2) for composites containing 218CS wire with each of the four alloy matrix materials is shown in figure 8. The data for only those specimens which failed in tension are plotted. The majority of the data obtained were for composites containing 218CS wire having a diameter of 0.008 inch (0.020 cm). Some stress-rupture data were also obtained for composites containing alloy 3 as the matrix and wires having diameters of 0.015 inch (0.038 cm) and 0.020 inch (0.051 cm) (fig. 8). It should be noted that composites containing alloy 3 and 218CS wires of 0.008- and 0.020-inch (0.020- and 0.05-cm) diameter were not fully densified, which could reduce its properties in stress-rupture. The results indicated that higher fiber contents are necessary to achieve a 100-hour rupture life at 2000° F (1093° C) and 15 000 psi (103.42 MN/m^2) for composites of alloys 1 and 5 than for alloys 3 and 7. Alloys 3 and 7 both contain titanium and aluminum additions. Alloy 3 appears to be slightly better than alloy 7 as a matrix material for the composites studied in stress-rupture. The composites having alloy 3 as the matrix and containing large-diameter fibers appear to be as strong as the composites containing the 0.008-inch (0.020-cm) fibers. The stress-rupture results on the fibers tested outside of

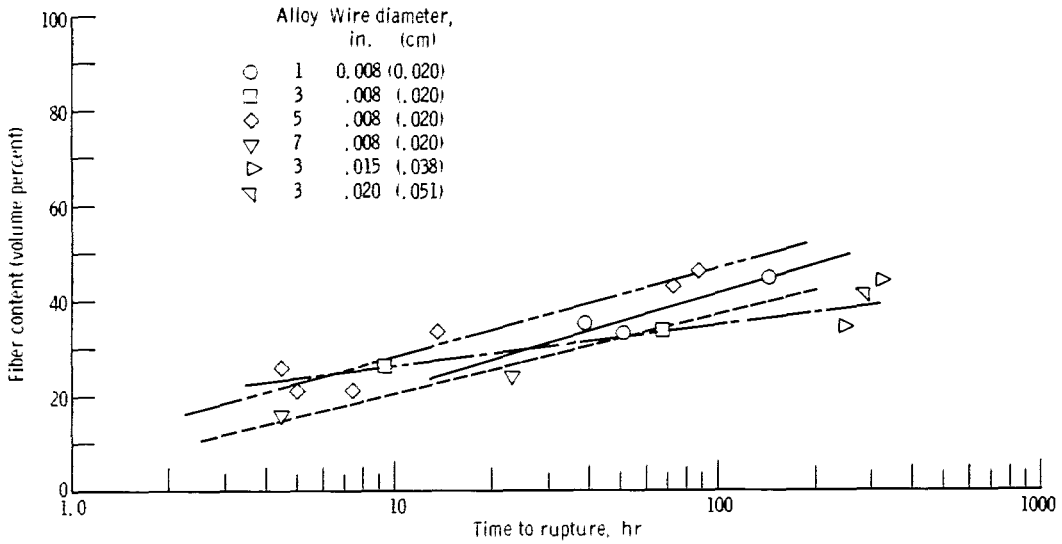


Figure 8. - Rupture properties of high-temperature fabricated composites at 15 000 psi (103.42 MN/m^2) and 2000° F (1093° C). Wire material, 218CS.

the composite, however, showed that the larger-diameter fibers were weaker than the 0.008-inch (0.020-cm) fibers.

Depth of Reaction Against Rupture Life

The properties reported for the different combinations of wire and alloy may be related to the degree of reactivity between the matrix and wire reinforcement as well as to the initial properties of these components of the composite. Generally, the smaller the depth of penetration into the fiber, the higher the composite properties. This simple gage of composite strength must be qualified for varying wire size and for variations in properties of the reacted zone. The depth of the reaction between the matrix and the fiber was measured for each specimen tested in stress-rupture. Figure 9 is a plot of the depth of reaction against time of exposure for composite specimens containing 218CS wire and tested at 2000° F (1093° C). On the basis of reaction depth after a 100-hour exposure at 2000° F (1093° C), composites having 0.015-inch (0.038-cm) diameter fibers and alloy 3 as the matrix appear to be the most compatible, having a penetration depth of only 1.3 mils (0.0033 cm). Alloy 7 also appears to be very compatible with the fibers having a depth of reaction of 1.6 mils (0.0041 cm) after 100-hour exposure. Composites containing 0.008-inch (0.020-cm) diameter fibers and alloy 3 as a matrix material show greater reaction with the fibers than composites that contained 0.015-inch (0.038-cm) diameter fibers. The composite specimens containing the smaller fibers, as mentioned

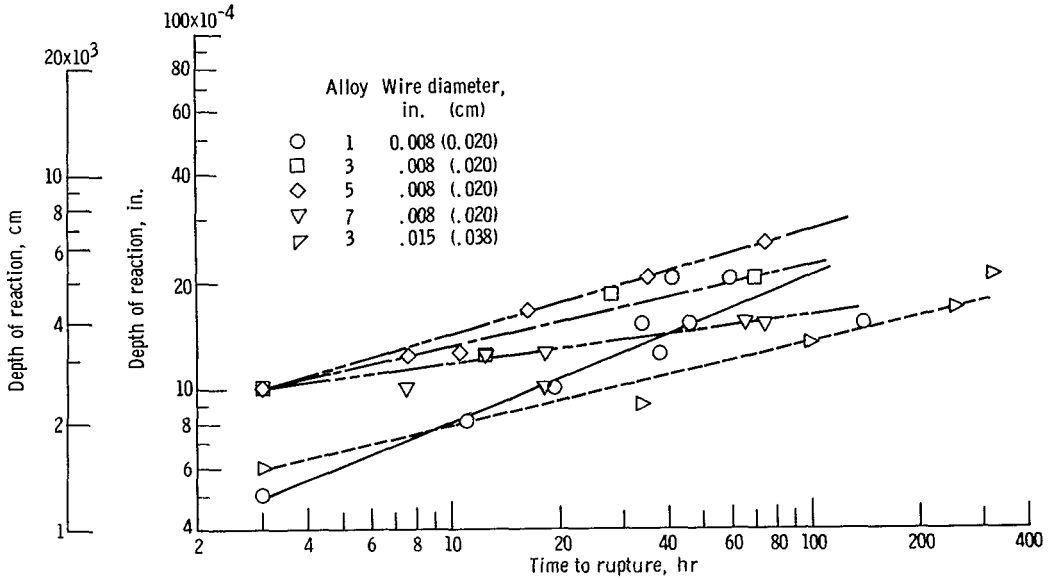


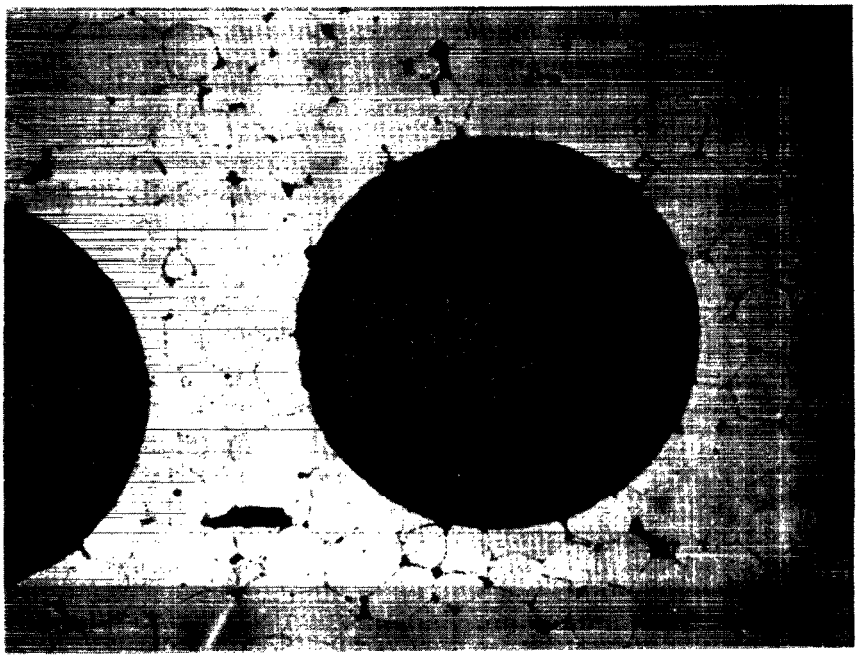
Figure 9. - Effect of time in rupture test at 2000° F (1093° C) on penetration depth of high temperature fabricated composites. Wire material, 218CS; all tests in helium atmosphere.

previously, were not fully densified. Another observation in this investigation was that the rate of reaction with the fiber was influenced by the porosity of the matrix material. The greater reaction rates during fabrication occurred for those composites in which the matrix was not fully densified, which indicates that surface diffusion may be controlling the reaction. Alloy 3 appeared to be the most compatible alloy used in this investigation based on the fully densified, large-diameter fiber composite data, followed by alloy 7. Both of these alloys contain titanium and aluminum additions and result in the strongest composites in stress-rupture. Diffusion coefficients calculated from the depth of penetration results varied between 2×10^{-11} to 5×10^{-11} square centimeter per second.

Optimization of Fabrication Technique and Wire Size

Fabricating fully densified composites, as was noted for the alloy 3 composite system, is a prerequisite to good compatibility and composite properties. In order to evaluate the stress-rupture properties of alloy 3 composites containing 0.008-inch (0.020-cm) diameter fibers, it was first necessary to obtain fully densified composites to limit surface diffusion at the final pressing temperature of 2000° F (1093° C). The fabrication procedure had been to sinter the slip-cast specimens at 2000° F (1093° C) for 1 hour in hydrogen and then to hot press the billet at 2000° F (1093° C) for 2 hours at 20 000 psi (137.89 MN/m^2) using pressurized helium. The fabrication procedure was modified in an attempt to densify the matrix powder as much as possible prior to densifying the material at 2000° F (1093° C). The slip-cast specimens were sintered at 1500° F (816° C) for 1 hour rather than 2000° F (1093° C) to drive off the binder and reduce oxide films present on the powders as well as to impart sufficient green strength to the casting. The sintered specimens were then hot pressed at 1500° F (816° C) for 1 hour at a gas pressure of 20 000 psi (137.89 MN/m^2). Figure 10 shows the microstructure of a typical as-pressed specimen. No penetration was observed, yet the matrix was fairly dense. The specimens were then hot pressed at 2000° F (1093° C) for 1 hour at a gas pressure of 20 000 psi (137.89 MN/m^2). Fully dense specimens were obtained by this technique, and the reaction due to fabrication was lowered.

It was noted in the section concerned with the stress-rupture properties of the composites that shear fractures occurred when the fibers were misalined which lowered the properties of the composite in stress-rupture. The maximum fiber content investigated was also approximately 50 volume percent. In order to assure better fiber alinement, smaller diameter cans were used than had been used previously, 0.375-inch (0.953-cm) inside diameter rather than 0.75-inch (1.91-cm) inside diameter. It was also attempted to increase the fiber content above 50 volume percent. Composites were fabricated using the modified sintering and pressing technique and using the smaller diameter cans.



Transverse section

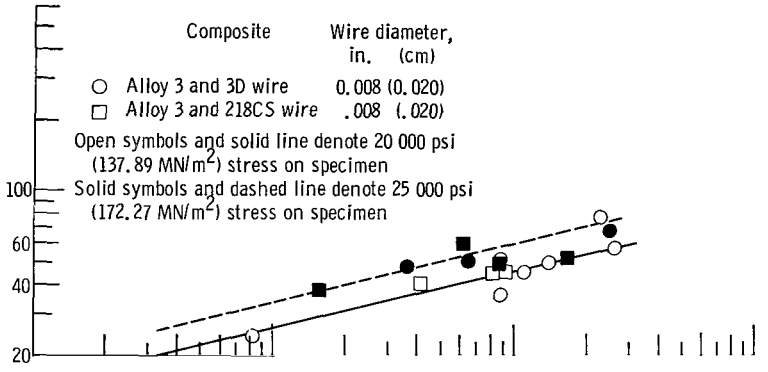
Longitudinal section

Figure 10. - Typical microstructure of low-temperature fabricated composites sintered at 1500° F (816° C) for 1 hour in hydrogen, pressed at 1500° F (816° C) for 1 hour; pressure, 20 000 psi (137.89 MN/m^2). X250.

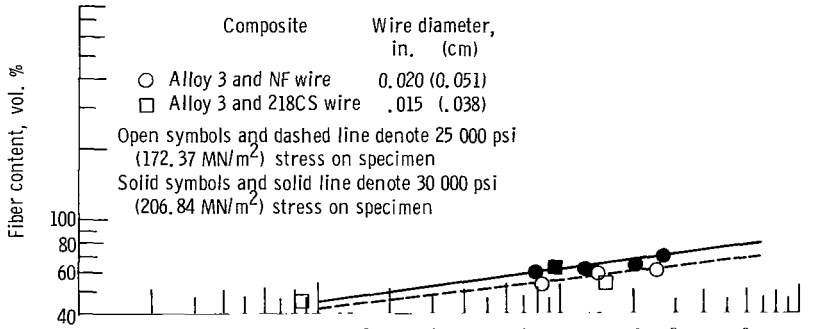
TABLE VIII. - STRESS-RUPTURE PROPERTIES OF ALLOY 3 COMPOSITES FABRICATED BY LOW-TEMPERATURE-DENSIFICATION TECHNIQUE

[All failures were of tensile type.]

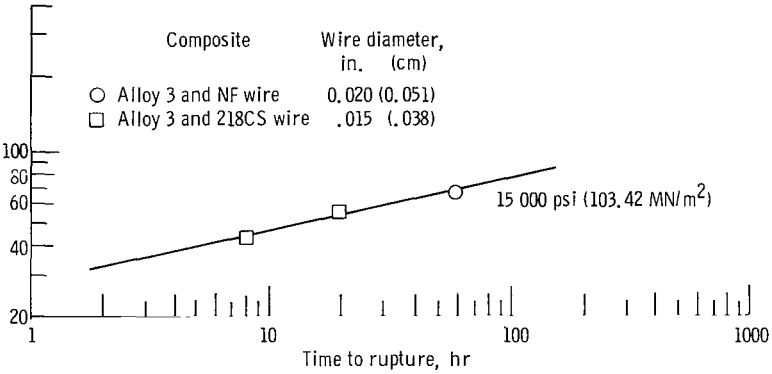
Wire material	Wire diameter		Stress		Life, hr	Fiber content, vol. %	Test temperature	
	in.	cm	psi	MN/m^2			^oF	^oC
3D	0.008	0.020	15 000	103.42	267.1	54.8	2000	1093
			20 000	137.89	8.2	24.1		
			↓	↓	88.2	36.3		
					109.7	45.4		
					139.7	49.6		
			22 000	151.69	95.2	49.5		
			25 000	172.37	35.7	47.6		
			25 000	172.37	65.6	49.7	↓	↓
218CS	0.008	0.020	20 000	137.89	41.1	40.8	2000	1093
			20 000	137.89	81.3	44.7		
			20 000	137.89	91.7	45.2		
			25 000	172.37	15.3	37.7		
			25 000	172.37	61.0	59.0		
			25 000	172.37	86.4	48.1	↓	↓
	0.015	0.038	25 000	172.37	8.5	44.8	2000	1093
			25 000	172.37	155.7	53.7		
			25 000	172.37	245.4	39.6		
			30 000	206.84	95.2	62.0	↓	↓
			15 000	103.42	7.8	43.5	2200	1204
			15 000	103.42	18.7	55.8	2200	1204
NF	0.020	0.051	25 000	172.37	84.2	52.8	2000	1093
			25 000	172.37	141.4	59.7		
			25 000	172.37	251.6	61.5		
			30 000	206.84	79.3	59.7		
			↓	↓	127.2	61.2		
					207.2	62.8		
					264.8	70.3	↓	↓
			15 000	103.42	57.9	66.6	2200	1204



(a) Alloy 3-218CS wire and alloy 3-3D wire composites tested at 2000° F (1093° C).



(b) Alloy 3-NF wire and alloy 3-218CS wire composites tested at 2000° F (1093° C).



(c) Alloy 3-NF wire and alloy 3-218CS wire composites tested at 2200° F (1204° C).

Figure 11. - Effect of fiber content in rupture life of low-temperature fabrication composites. All tests in helium atmosphere.

TABLE IX. - REACTION DEPTH AND EXPOSURE TIME

FOR ALLOY 3 COMPOSITES

[Fabricated by low-temperature-densification technique.]

Wire material	Wire diameter		Reaction depth		Exposure time, hr	Test temperature	
	in.	cm	in.	cm		°F	°C
3D	0.008	0.020	0.00025	0.00064	1.0	2000	1093
			.00045	.00114	9.2		
			.00100	.00254	36.7		
			.00091	.00231	66.6		
			.00100	.00254	89.2		
			.00100	.00254	96.6		
			.00130	.00330	110.7		
			.00180	.00457	140.7		
			.00140	.00356	268.1		
218CS	0.008	0.020	0.00050	0.00127	1.0	2000	1093
			.00075	.00191	16.3		
			.00100	.00254	42.1		
			.00150	.00381	62.0		
			.00125	.00318	82.3		
			.00125	.00318	87.4		
			.00125	.00318	92.7		
	0.015	0.038	0.00037	0.00094	1.0	2000	1093
			.00060	.00152	9.5		
			.00123	.00312	96.2		
			.00125	.00318	156.7		
			.00170	.00432	245.4		
			.00170	.00432	7.8	2200	1204
			.00170	.00432	18.7	2200	1204
NF	0.020	0.051	0.00063	0.00160	1.0	2000	1093
			.00180	.00457	80.3		
			.00250	.00635	85.2		
			.00250	.00635	128.2		
			.00250	.00635	141.4		
			.00260	.00660	252.6		
			.00270	.00686	265.8		
			.00310	.00787	207.2		
			.00500	.01270	57.9	2200	1204

The results obtained with composites containing 0.008-, 0.015-, and 0.020-inch (0.020-, 0.038-, and 0.051-cm) diameter wire using alloy 3 as the matrix material are tabulated in table VIII. Stress-rupture tests were conducted at 2000° F (1093° C) and in some cases at 2200° F (1204° C). It should be noted that all the composite specimens failed in tension and that higher fiber contents were obtained particularly with composites containing the larger diameter fibers. A plot of fiber content against rupture life for composites containing 3D and 218CS wire (0.008-in. (0.020-cm) diam) is shown in figure 11(a). The data plotted are for composites stressed at 20 000 and 25 000 psi (137.89 and 172.37 MN/m^2) at 2000° F (1093° C). The properties of both these composite systems as a function of fiber content appear to be independent of wire composition. Figure 11(b) is a plot of fiber content against rupture life for composites of alloy 3 containing 0.020-inch (0.051-cm) diameter NF wire and for composites containing 0.015-inch (0.038-cm) diameter 218CS wire. The properties of both systems as a function of fiber content also appear to be independent of wire composition. A plot of fiber content against rupture life for composites containing the large-diameter fibers which were tested at 2200° F (1204° C and 15 000 psi (103.42 MN/m^2) is shown in figure 11(c). The results indicate that at a stress of 15 000 psi (103.42 MN/m^2) approximately 75-volume-percent fiber is necessary to achieve a 100-hour rupture life.

The reaction between the matrix and fiber was measured for each specimen tested in stress-rupture and is shown in table IX. Figure 12 is a plot of the depth of penetration as a function of rupture time for alloy 3 composites containing either 0.008- or

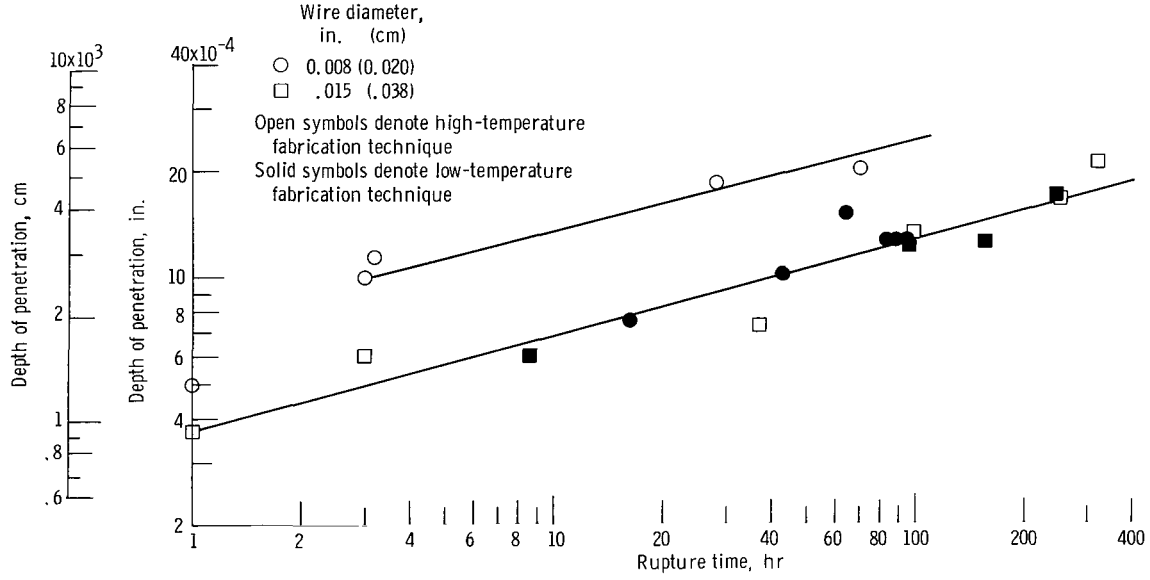
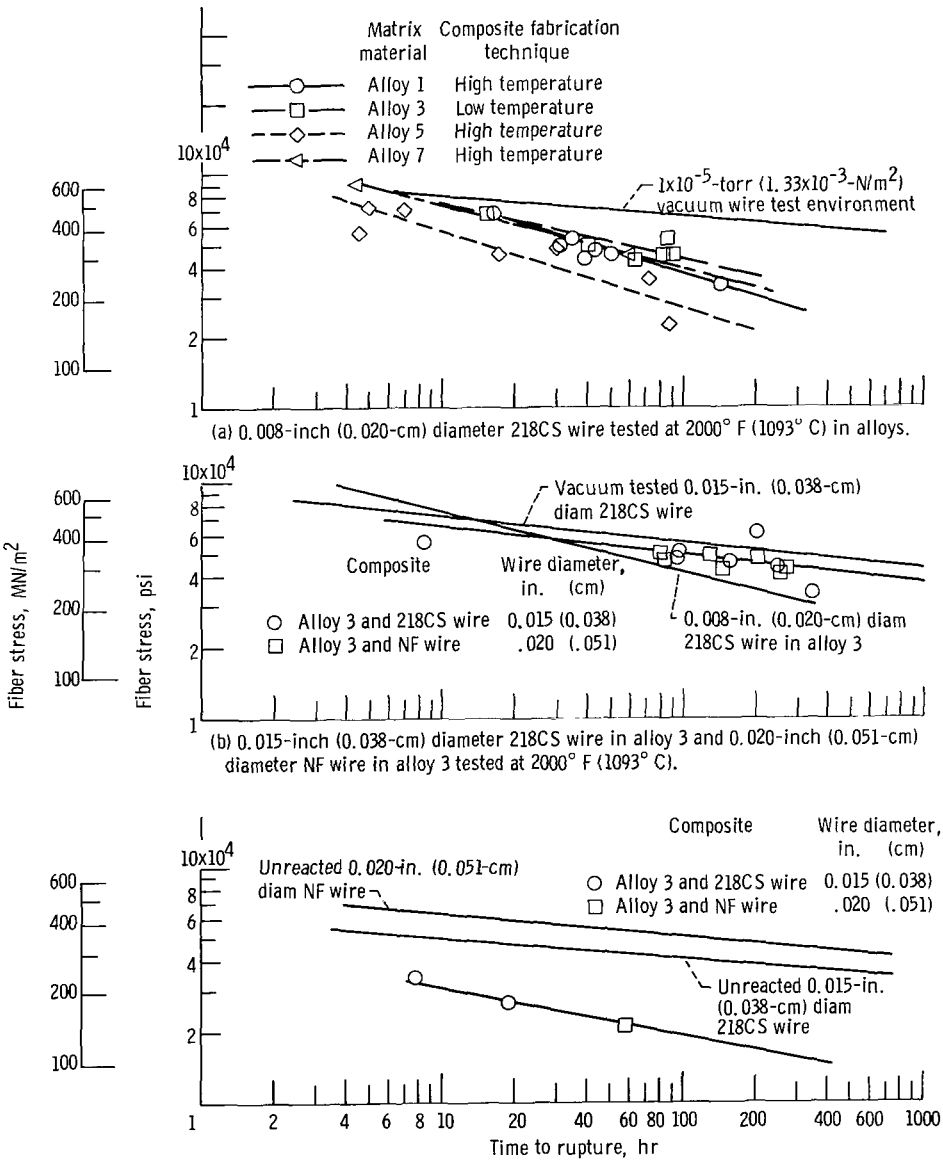


Figure 12. - Effect of fabrication process and wire diameter on depth of penetration in rupture test at 2000° F (1093° C). All tests in helium atmosphere; composite, alloy 3 and 218CS wire.

0. 015-inch (0. 020- or 0. 038-cm) diameter fibers fabricated by both the high-and low-temperature-densification techniques. For the rupture time intervals plotted, the low-temperature-densification technique did not change the rate of reaction between the fiber and matrix for the large-diameter fiber composites. The depth of penetration values obtained for composites having the smaller-diameter fibers and prepared by the modified technique were, however, much less than those obtained using the initial fabrication technique, as shown in the plot. It can be seen from table IX that the depth of penetration as a function of rupture time is approximately equivalent for the 3D wire and for the 218CS wire, but the NF wire has reaction depths approximately twice those of the other two wires studied.

Since the composite systems studied were tested at different stresses and contained varying fiber contents, it is difficult to evaluate which matrix and wire combinations were the strongest. If it is assumed that the fiber carries the major portion of the load during stress-rupture and that the matrix contribution is small, which is a valid assumption based on the results obtained in reference 4, then the stress on the fiber contained in a composite can be calculated as a function of rupture life. The stress-carrying capabilities of the wire in the different matrix materials can then be evaluated, and a determination of the best wire-matrix combination can be made. The stress on the fiber was calculated from the composite stress-rupture results as a function of rupture time and is plotted in figure 13(a) for 0. 008-inch (0. 020-cm) diameter 218CS wire. The stress on the matrix was neglected, and the specimen load was divided by the fiber area contained in the composite. The data used for the stress on 0. 008-inch (0. 020-cm) diameter 218CS wire were taken from alloy 3 matrix composites fabricated using the modified technique. Alloy 3 appears to be the best matrix alloy for stress-rupture at 2000° F (1093° C). Approximately 65-percent of the properties of the wire are retained in the composite for a rupture life of 100 hours. Figure 13(b) is a plot of the stress on the fiber against rupture life for composites containing the large-diameter fibers. Data for the stress on an unreacted fiber and for the fiber from composites of alloy 3 containing 0. 008-inch (0. 020-cm) diameter fibers are also plotted for comparison. The stress contribution of the 218CS wire and NF wire appear to be equivalent. Approximately 90 percent of the properties of the 218CS wire is retained in the composite for rupture in 100 hours. It can be seen that the stress to cause rupture for times exceeding 30 hours is higher for the large-diameter fibers than for the smaller-diameter fibers. For short-time applications, reinforcement with the smaller-diameter fibers is superior to large-diameter-fiber reinforcement, but, for long-time applications, the larger diameter fibers are superior. The plot can also be used to determine the stress-rupture properties of composites containing varying volume fractions of fibers. The stress on the fibers to cause rupture in a specific time is multiplied by the volume fraction of fiber contained in the composite. From the data shown in the plot, for



(a) 0.008-inch (0.020-cm) diameter 218CS wire tested at 2000° F (1093° C) in alloys.

(b) 0.015-inch (0.038-cm) diameter 218CS wire in alloy 3 and 0.020-inch (0.051-cm) diameter NF wire in alloy 3 tested at 2000° F (1093° C).

(c) 0.015-inch (0.038-cm) diameter 218CS wire in alloy 3 and 0.020-inch (0.051-cm) diameter NF wire in alloy 3 tested at 2200° F (1204° C).

Figure 13. - Rupture properties of wire in composites compared with wire tested in vacuum.

example, it would be expected that a composite containing the larger-diameter fibers and having a fiber content of 70 volume percent would have a 100-hour stress-rupture strength of 35 000 psi (241.32 MN/m^2)(0.70×50 000 psi (344.74 MN/m^2)) at 2000° F (1093° C). Figure 13(c) is a plot of the stress on the large-diameter fibers contained in composites of alloy 3 and tested at 2200° F (1204° C). The properties in stress-rupture of the unreacted fibers is also shown. A much greater loss in the fiber properties in the composite occurs at this temperature than at 2000° F (1093° C). Less than 50 percent of the properties of the 218CS fiber is retained in the composite for a rupture life of 100 hours. Less than 40 percent of the properties for the reacted NF wire is retained.

DISCUSSION

Composites were produced having stress-rupture properties superior to conventional superalloys at use temperatures of 2000° and 2200° F (1093° and 1204° C). Figure 14 is a plot of the stress to cause rupture in 100 and 1000 hours against test temperature for alloy 3 composites containing 70-volume-percent large-diameter (0.015-in. (0.038 (0.038-cm) diam 218CS and 0.020-in. (0.051-cm) diam NF) fibers as compared with

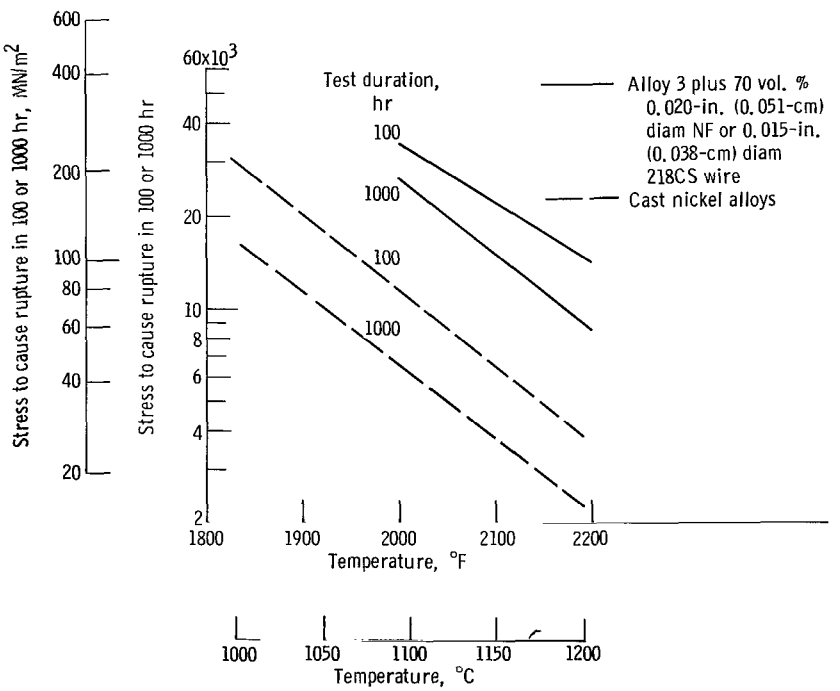


Figure 14. - Stress for rupture in 100 and 1000 hours of 70-volume-percent composites and nickel alloys.

cast nickel-base alloys, such as M22VC (ref. 11) and NASA-TRW-VI-A (ref. 12). The 100-hour stress-rupture strength obtainable for the composite at 2000° F (1093° C) is 35 000 psi (241.32 MN/m^2) as compared with 11 500 psi (79.29 MN/m^2) for the best cast nickel alloys. At 2200° F (1204° C) the 100-hour stress-rupture strength obtainable for the composite was 14 000 psi (96.53 MN/m^2) as compared with 4000 psi (27.58 MN/m^2) for the cast nickel alloys. The 100-hour rupture strength for the composite at 2000° F (1093° C) represents a use temperature advantage over cast nickel alloys of approximately 200° F (93° C). Data were not obtained at a stress level of 35 000 psi (241.32 MN/m^2) for the composite specimens tested at 2000° F (1093° C) because of the limited number of specimens produced with high-volume-percent fiber contents. Lower stress levels were used for specimens with lower fiber contents to give tests with reasonably long rupture lives in order to evaluate the reaction between the fiber and matrix material after long exposure times. Data were obtained for such composites in which the stress on the fiber, the strength controlling component, was over 50 000 psi (344.74 MN/m^2) and exposure time was over 300 hours. The depth of penetration and fiber stress-rupture data were obtained with more easily produced, lower-fiber-content billets. Specimens of 70-volume-percent fiber contents can be reporduced with the techniques evolved in this investigation, in fact, specimens with over 75-volume-percent fiber contents can be made. As shown in the RESULTS section, the data obtained in this investigation can be used to determine the 100- and 1000-hour stress-rupture strength of composites containing 70-volume-percent fiber contents.

The density of the composite material is much greater than that of the nickel alloy and must be taken into consideration. The tensile stresses in turbine blades, for example, are a result of centrifugal loading; therefore, the density of the material is important. Tungsten has a density about 2.3 times that of most nickel-base alloys, and a composite containing 70-volume-percent tungsten fibers has a density approximately 1.9 times that of most nickel-base alloys. On a specific strength basis, the temperature advantage of the composite is thus reduced. Figure 15(a) is a plot of the ratio of stress to cause rupture to density (specific rupture strength) against time to rupture for alloy-3 composites reinforced with both 50- and 70-volume-percent wire compared with unreinforced alloy 3 and cast nickel alloys at a test temperature of 2000° F (1093° C). Both 20-mil (0.051-cm) NF wire and 15-mil (0.038-cm) 218CS wire give the same result. The 70-volume-percent reinforced composite is more than 5 times as strong for a 100-hour rupture life than for the unreinforced alloy 3, based on a specific strength consideration. A comparison was also made with cast nickel alloys. The 70-volume-percent fiber reinforced composite is approximately 60 percent better than the cast nickel alloys for rupture in 100 hours and 3 times as strong for rupture in 1000 hours. The same type of plot and comparisons are shown in figure 15(b) for specimens tested at 2200° F (1204° C). The 70-volume-percent fiber reinforced composite

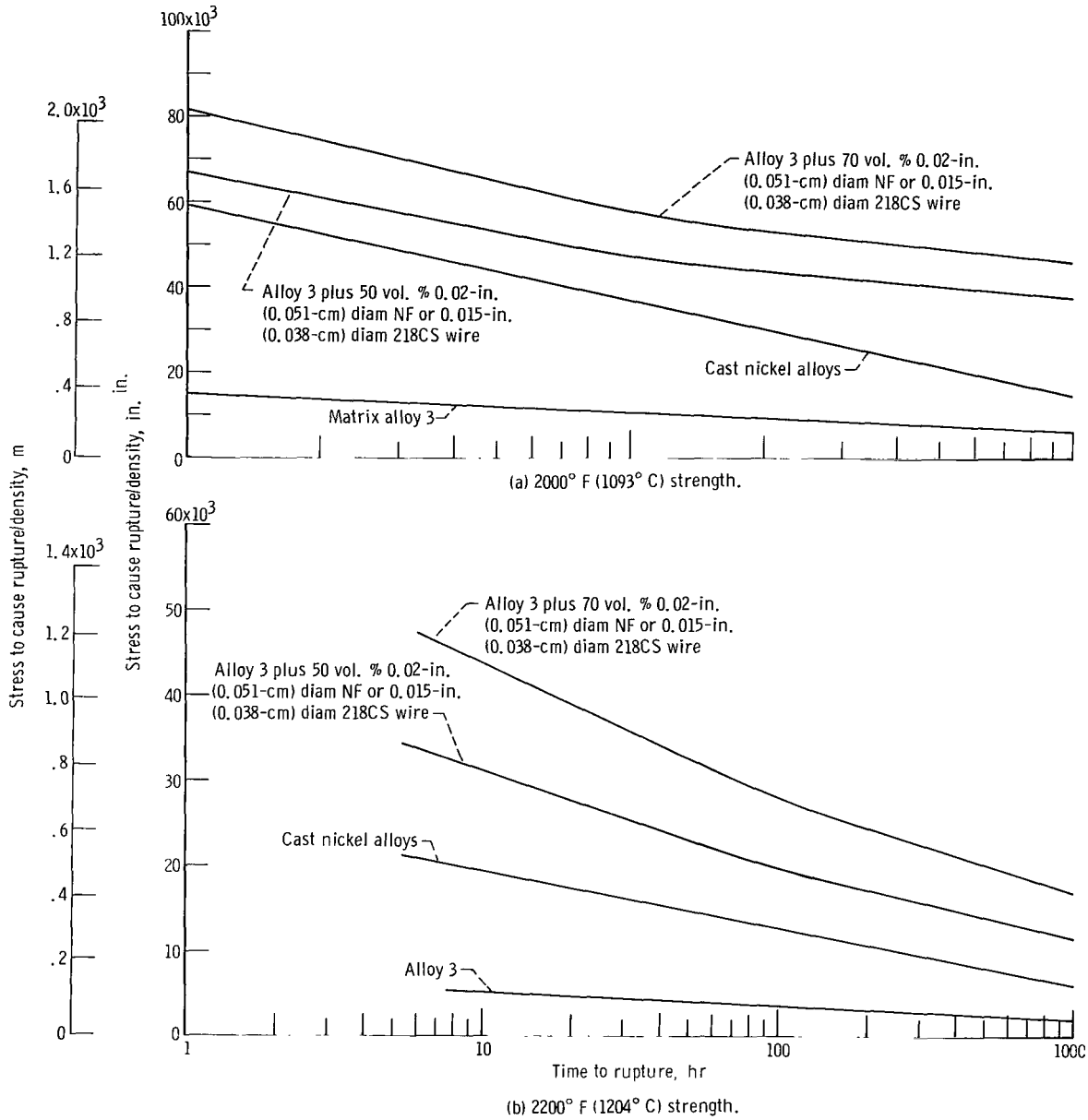


Figure 15. - Comparison of specific rupture strength of 50- and 70-volume-percent composites; cast nickel alloys and matrix alloy 3.

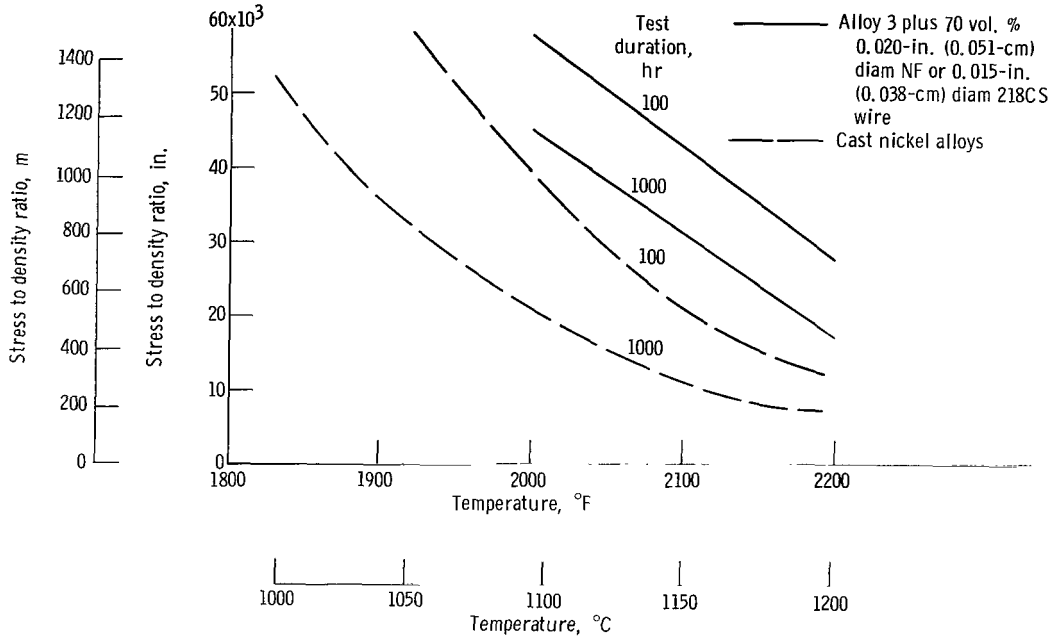


Figure 16. - Stress to density ratio for rupture in 100 and 1000 hours of 70-volume-percent composites and cast nickel alloys.

is 2 times as strong as the cast nickel alloys for 100-hour rupture life and 2.5 times as strong for an expected 1000-hour rupture life.

The specific strength for rupture in 100 and 1000 hours against test temperature is plotted in figure 16 for a composite reinforced with 70-volume-percent fiber and for cast nickel alloys. The advantage of the composite increases with temperature from 2000° to 2200° F (1093° to 1204° C). At 2000° F (1093° C) the stress to density ratio of the composite for rupture in 100 hours is 57.5×10^3 inches (1460.5 m). At the same strength to density ratio cast nickel alloys fail in rupture at 100 hours at 1925° F (1052° C). The temperature advantage of the composite at this particular strength to density ratio and rupture time is thus 75° F (24° C). If the stress to density ratio for rupture in 100 hours at 2200° F (1204° C) for the composite is considered, then a use temperature advantage of 140° F (60° C) is obtained. For long-time applications, the composite shows even better use temperature advantage over cast nickel alloys. The use temperature advantage for rupture in 1000 hours for the composite, for example, is 130° F (54° C), based on its 2000° F (1093° C) strength, and 170° F (77° C), based on its properties at 2200° F (1204° C).

Alloying Effect on Properties

Composite strength can be related to the compatibility with the matrix material. Stronger composites were produced with matrix materials which reacted less with the fibers than those which were less compatible with the fibers. Alloy 5, which was found to be the most reactive material with the fiber, also resulted in the poorest composite properties, but alloy 3, which was found to be the least reactive matrix material, produced the strongest composites in stress-rupture.

Nickel alloys that contained titanium and aluminum additions (alloys 3 and 7) appeared to be more compatible with the fibers investigated than nickel alloys that did not contain these additives (alloys 1 and 5). The reaction between the mutually soluble fiber and matrix materials was limited to approximately 1.25 mils (0.00318 cm) after exposure for 100 hours at 2000° F (1093° C). Higher composite strength might be obtained by further modification of the matrix composition.

The refractory-wire composition also influences the compatibility between the fiber and matrix. The 218CS and 3D (tungsten - 3-percent rhenium) wires were more compatible with the nickel alloys investigated than were NF (tungsten - 1-percent thoria) or TZM (a molybdenum alloy) wires. The reaction with NF wire was twice as great as with the 218CS or 3D wire for a 100-hour exposure at 2000° F (1093° C). The TZM wire completely reacted with the nickel alloys during fabrication. The strength retention of wires having the better compatibility was greater than those fibers having poorer compatibility.

Optimization of Wire Size

The fiber contribution to the composite strength can be increased if the effect of loss in strength of the fiber due to alloying reactions can be decreased. To do this, one must limit the reaction with the fiber or limit the effect of the reaction on the fiber strength contribution to the composite. The effect of the alloying reaction on fiber strength can be reduced by varying the wire diameter of the reinforcing material. Because the decrease in fiber strength with time governs the stress-rupture properties of the composite, it is necessary to limit this decrease. The rate of penetration of the matrix into the fiber would be expected to be nearly constant regardless of the diameter of the fiber used. The fraction of fiber area reacted with time, however, would be less as the fiber diameter is increased. If, for example, at the end of 100 hours exposure at a specific temperature, the depth of penetration into the wire is 2 mils (0.005 cm), then 75 percent of an 8-mil (0.20-cm) wire would have been reacted, while only 36 percent of a 20-mil (0.051-cm) diameter fiber would have been reacted. Smaller diameter wire, however, is generally stronger than larger diameter wire so that both factors must be con-

sidered as they affect the strength of the reacted fiber. A graphical technique was used to illustrate schematically the variation of composite strength as a function of wire size and depth of reaction of the wire with the matrix. It is assumed that, for a specific rupture life, the stress-rupture properties of the alloyed zone of the fiber is constant for all wire diameters and that the unreacted portion of the fiber carries a stress at the specific rupture time equal to that of a fiber which has not been reacted. It is also assumed that the stress carrying capabilities of the fiber is proportional to the volume fraction of the two zones present in the fiber. Using these assumptions, the plot shown in figure 17(a) can be constructed. The stress on the reacted fiber for a rupture life of 100 hours is plotted against the reaction depth of the fiber for varying wire diameter. The plot shows that, for reaction depth less than approximately 1 mil (0.0025 cm), the 8-mil (0.020-cm) wire is stronger than the other wire sizes but that, at greater

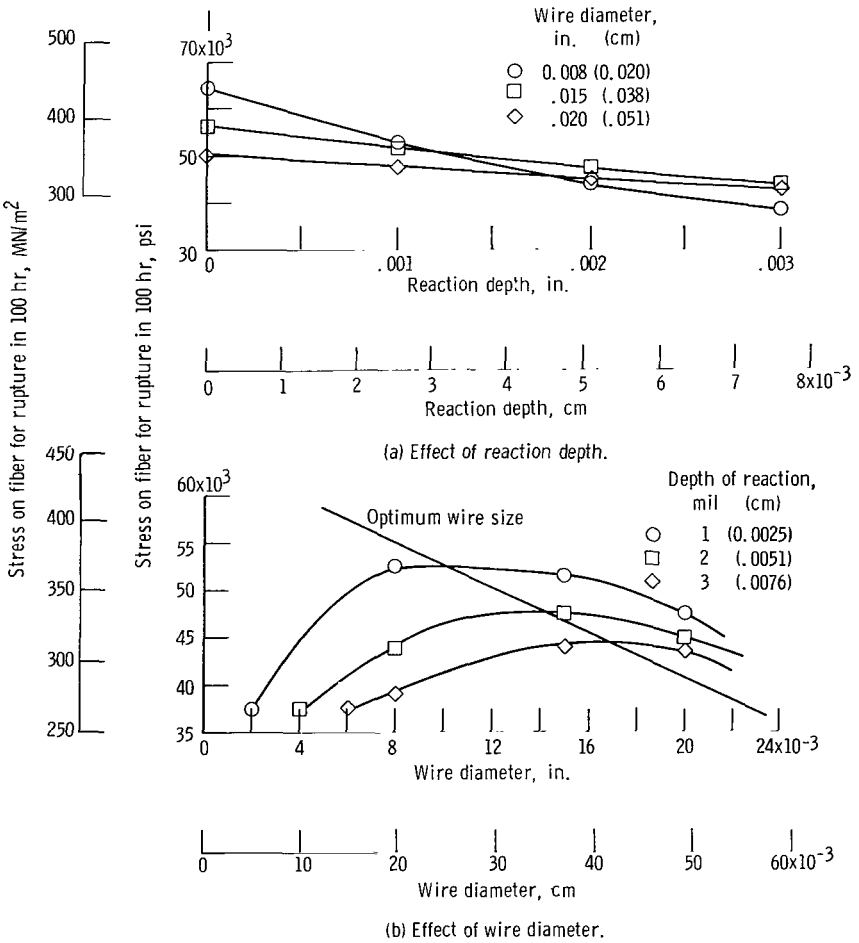


Figure 17. - Calculated 100-hour rupture strength of 218CS wire at 2000° F (1093° C) as function of wire diameter and depth of penetration.

reaction depths, the larger diameter fibers are stronger. This result is consistent with the result obtained on composites containing various diameter fibers, in that, for short-time applications where the depth of reaction was small, the smaller diameter fiber-containing composites were stronger, and, for long-time applications, the larger-diameter fiber-containing composites were stronger. The plot shown indicates that there is an optimum wire size, dependent on the depth of reaction, which should give the best strength contribution to the composite. Figure 17(b) is a plot of the stress on the reacted fiber for rupture in 100 hours against wire diameter as a function of varying reaction depths. The straight line drawn through the curves is an approximation of the optimum wire size for use at the specific reaction depths after exposure for 100 hours at 2000° F (1093° C). The optimum wire size is that wire diameter which will yield the highest fiber stress contribution to the composite at a specific reaction depth. Figure 18 is a plot of the optimum wire diameter against the depth of penetration or reaction. Reaction depths of approximately 1.3 mils (0.0033 cm) after exposure for 100 hours were obtained for composites having 218CS wire as the reinforcment. The optimum wire diameter for rupture in 100 hours is approximately 11 mils (0.028 cm) based on the assumptions used to construct the plot. Although 11-mil (0.028-cm) diameter wire was not used in this investigation to varify the validity of this technique, the results of the data obtained in this study indicate that the optimum wire size for 100 hour stress-rupture at 2000° F (1093° C) is between 8 and 15 mils (0.020 and 0.038 cm). The technique is presented only to show that the wire diameter of the reinforcing fiber must be taken into consideration when designing a composite in which reaction with the matrix occurs. An inherent assumption in the preceeding discussion is that the larger diameter wires are sufficiently long to preclude shear-pullout of the fiber. Increasing

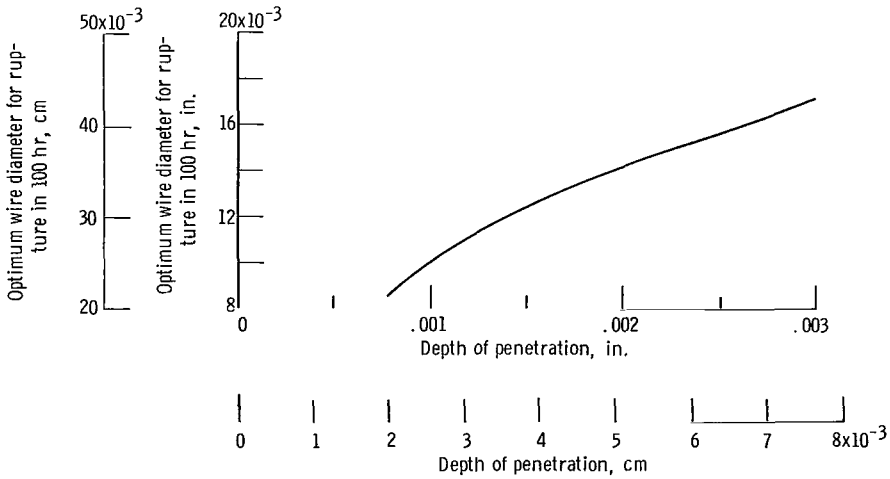


Figure 18. - Effect of depth of penetration on optimum wire diameter.

diameter can reduce the effective strength of fiber composites as the fiber length approaches the critical length.

CONCLUDING REMARKS

Substantial improvements in stress-rupture strength have been achieved by reinforcing nickel-base alloys with tungsten wires. On a specific strength basis, this gain is reduced owing to the high density of the tungsten wires. Nonetheless, the composite material is superior to current nickel-base alloys for use at 2000° and 2200° F (1093° and 1204° C). Composites containing up to 70-volume-percent fiber were fabricated using the procedures developed. The fabrication procedure and the composition of the alloy matrix materials used limited the extent of the reaction with the fiber to approximately 1.25 mils (0.0032 cm) in 100 hours at 2000° F (1093° C). The high-volume-percent fiber contents and limited matrix reaction with the fiber produced tungsten-fiber reinforced nickel alloy composites with properties at 2000° and 2200° F (1093° and 1204° C) superior to those of the best nickel-base alloys available. These results thus indicate that the reactivity between refractory fibers and nickel-base alloys can be minimized to obtain strong fiber reinforced composites for use at 2000° F (1093° C) and above. The information gained concerning the degree of reactivity between the fiber and matrix would serve to guide subsequent investigations to obtain even stronger composites than those of the current investigation. These stronger composites would utilize improved refractory-metal wires currently under development.

CONCLUSIONS AND SUMMARY OF RESULTS

This investigation was conducted to produce fiber reinforced superalloy composites having stress-rupture properties superior to conventional superalloys at use temperatures of 2000° and 2200° F (1093° and 1204° C). It was also intended to determine whether variations in the matrix composition would promote compatibility between the matrix and the fiber to enhance composite strength. The results obtained and the conclusions based on them are as follows:

1. Composites were produced having stress-rupture properties superior to conventional superalloys at use temperatures of 2000° and 2200° F (1093° and 1204° C). The 100-hour stress-rupture strength obtained for the composite at 2000° F (1093° C) was 35 000 psi (241.32 MN/m^2) as compared with 11 500 psi (79.29 MN/m^2) for the best cast nickel alloys. At 2200° F (1204° C), the 100-hour stress-rupture strength for the composite was 14 000 psi (96.53 MN/m^2) as compared with 4000 psi (27.58 MN/m^2) for cast

nickel alloys. The use temperature of the composite based on density considerations was 140° F (60° C) higher than the best cast nickel alloys for specific strength to cause rupture in 100 hours and 170° F (77° C) higher for specific strength to cause rupture in 1000 hours.

2. Composite strength is related to the compatibility of the reinforcing fiber with the matrix material. The greater the reaction between the matrix and the fiber, the lower the strength properties of the composite.

3. Nickel alloys that contain titanium and aluminum additions were more compatible with the fibers investigated than nickel alloys that did not contain these additives. In those composites containing these nickel alloys, the reaction between the mutually soluble fiber and matrix materials was limited to approximately 1.25 mils (0.0032 cm) after exposure for 100 hours at 2000° F (1093° C). This suggests that compatibility between the fiber and the matrix might be further improved if the precentages of these and other additives could be optimized. Higher composite strength would thus be obtained.

4. The refractory-wire composition also influences the compatibility between the fiber and matrix. The 218CS and 3D (tungsten-3 percent rhenium) wire were more compatible with the nickel alloys investigated than were the NF (tungsten-1-percent thoria) or TZM (a molybdenum alloy) wire. The reaction with NF wire was twice as great as that with 218CS or 3D wire for the 100-hour exposure at 2000° F (1093° C). The TZM wire completely reacted with the nickel alloys during fabrication.

5. Wire diameter is improtant to the design of composites in which reaction between the fiber and matrix material occurs. The strength contribution of the reacted fiber in a composite can be related to the area fraction of the fiber that has been alloyed. In this study, the strength contribution of the fiber decreased as the area fraction of the alloyed portion of the fiber increased. As the fiber diameter increased, however, the unalloyed fiber strength generally decreased. A technique has been developed that takes into account both factors and that permits the prediction of composite strength as a function of wire size and compatibility with the matrix.

6. For short-time applications, small diameter fibers were more advantageous than large diameter fibers. For long-time applications, however, large diameter fibers were superior. For example, the stress-rupture properties of the larger diameter fibers contained in a composite were reduced only 10 percent for rupture in 100 hours at 2000° F (1093° C), as compared with unreacted fibers tested outside of the composite at the same temperature. The stress-rupture properties of smaller-diameter fibers contained in composites were reduced over 30 percent for rupture in 100 hours at 2000° F (1093° C,, as compared to fibers tested outside of the composite.

7. A linear relation was found to exist between fiber content and rupture life at a constant stress level and temperature for composite systems studied in this investigation.

8. The powder metallurgy technique used in this investigation were capable of producing fully densified fiber reinforced superalloy composites containing up to 70-volume-percent fiber contents.

Lewis Research Center,
National Aeronautics and Space Administration,
Cleveland, Ohio, May 29, 1968,
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